Electronic Supplementary Material (ESI) for ChemComm.
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Supporting Information

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1. General information

^{1}H NMR spectra were recorded on commercial instruments (400 MHz). Chemical shifts were reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$, δ = 7.26). Spectra were reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration and assignment. ^{13}C NMR spectra were collected on commercial instruments (100 MHz) with complete proton decoupling. Chemical shifts are reported in ppm from the tetramethylsilane with the solvent resonance as internal standard ($CDCl_3$, δ = 77.0).

Enantiomeric ratio (*e.r.*) were determined by HPLC analysis using the corresponding commercial chiralpak column as stated in the experimental procedures at 25 ^{o}C. The chiralpak column Lux 5u Cellulose-2 and the chiralpakLux 5u cellulose-3 were purchased from phenomenex company.

Optical rotations were reported as follows: $[a]_D^{25}$ (*c*: g/100 mL, in solvent).

HRMS was recorded on a commercial apparatus (ESI Source).

All catalytic reactions were run in dried glassware or test tube.

THF, toluene and diethyl ether (Et_2O) were distilled from sodium and benzophenone as indicator.

CH_3CN and CH_2Cl_2 was distilled over CaH_2.

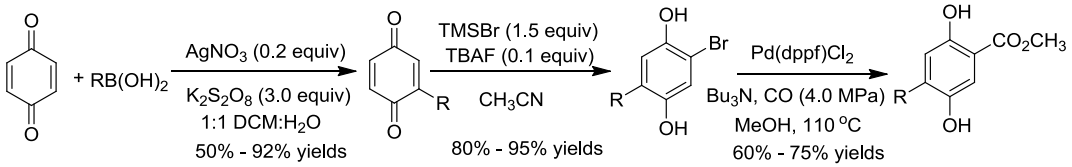
$Cu(OTf)_2$ (99%), Ag_2O were purchased from Alfa.

All racemic products (**3aa**-**3ua, 3ab**-**3ao**) were obtained by using $Cu(OTf)_2$ (10 mol%) and racemic *N*,*N*'-dioxide ligand (**L-RaPr$_2$** or **L-RaPr$_3$**, 10 mol%) as the catalyst according to general procedure for the catalytic enantioselective quinone – fulvene [2 + 2] cycloadditions.

Quinone substrates (**1a**, **1g**) were synthesized by using the literature method (J. Jacobs, S. Claessens, B. M. Mbala, K. Huygen, N. D. Kimbe, *Tetrahedron*, **2009**, *65*, 1193-1199).

Fulvenes were obtained by using the literature method (K. J. Stone, R. D. Little, *J. Org. Chem*, **1984**, *49*, 1853-1857; I. Erden, F. P. Xu, A. Sadoun, W. Smith, G. Sheff, M. Ossun, *J. Org. Chem.* **1995**, *60*, 813-820).

2. The method for the synthesis of quinone substrates [1]

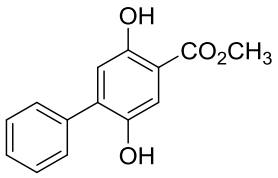


To a solution of benzoquinone (20 mmol, 1.0 equiv) in dichloromethane (100 mL) was added the corresponding boronic acid (30 mmol, 1.5 equiv), water (100 mL), and silver(I) nitrate (680 mg, 4.0 mmol, 0.2 equiv). Potassium persulfate (16.2 g, 60 mmol, 3.0 equiv) was then added and the solution was stirred vigorously at room temperature and monitored by thin-layer chromatography analysis of the organic layer. Upon consumption of quinone (3 – 24 h), the reaction was diluted with dichloromethane (50 mL). The layers were separated, and the aqueous layer was extracted with dichloromethane (3 x30 mL), dried over sodium sulfate, and evaporated in vacuo. The product was used for next step with silica gel quick purification.

To a solution of corresponding substituted benzoquinone in CH_3CN (30 mL), trimethylsilylbromide (TMSBr, 30 mmol, 4.0 mL) was added, and tetraethylammonium fluoroborate (TBAF, 1.0 M in THF, 1.5 mmol, 1.5 mL) in CH_3CN (10 mL) was carefully added, at which time the quinone color was disappeared. The reaction mixture was stirred for 2 hours at room temperature. After removal of solvent in vacuo, the resulted mixture was mixed with water (10 mL) and ethyl acetate (30 mL).The aqueous layer was separated and extracted with ethyl acetate twice (2 x 15 mL). The organic layers were combined and dried over sodiumsulfate, filtrated and removal of the solvent in vacuo. Purification was performed by silica gel chromatography to yield the pure product.

To a solution of the corresponding bromo-substituted benzene-1,4-diol (10 mmol) in DMF (15 mL), $Pd(dppf)Cl_2$ (0.75 g, 1.0 mmol), MeOH (6 mL), Bu_3N (2.74 mL, 14.8 mmol) was added in sequence. The vessel was purged and pressurized with CO (4.0 MPa) and stirred at 110 °C for 24 h. The reaction was cooled to room temperature, diluted with CH_2Cl_2 (50 mL), washed with 1N HCl (2 x 35 mL), brine/water (1:1, 35 mL), and brine (35 mL). The organic layers were combined, dried over Na_2SO_4, and concentrated under reduced pressure to afford a dark purple oil. The residue was purified by flash chromatography (5-10% EtOAc/hexanes) to afford the titled compound as a white solid.

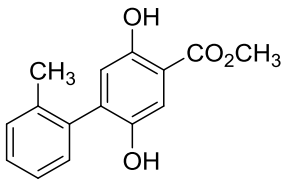
1. a) Y. Fujiwara, V. Domingo, I. B. Seiple, R.Gianatassio, M. D. Bel, and P. S. Baran, *J. Am. Chem. Soc.* **2011**, *133*, 3292-3295; b) M.Nakazaki, K.Naemura,*J. Org. Chem.* **1981**, *46*, 106-111; c) L. L. Miller, R. F. Stewart, *J. Org. Chem.* **1978**, *43*, 3078-3079; d) D. A. Evans, J. M. Wu, *J. Am. Chem. Soc.* **2003**, *125*, 10162-10163.



Methyl-2,5-dihydroxy-[1,1'-biphenyl]-4-carboxylate

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.31 (s, 1H), 7.55 – 7.39 (m, 6H), 6.90 (s, 1H), 4.82 (s, 1H), 3.95 (s, 3H);

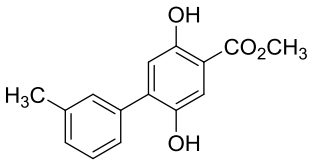
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.1, 155.4, 144.8, 136.5, 135.9, 129.3, 128.7, 128.7, 118.6, 115.6, 111.9, 52.4.



Methyl-2,5-dihydroxy-2'-methyl-[1,1'-biphenyl]-4-carboxylate

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.34 (s, 1H), 7.43 (s, 1H), 7.34 – 7.31 (m, 2H), 7.29 – 7.26 (m, 1H), 7.20 (d, *J* = 7.2 Hz, 1H), 6.78 (s, 1H), 4.48 (s, 1H), 3.96 (s, 3H), 2.18 (s, 3H);

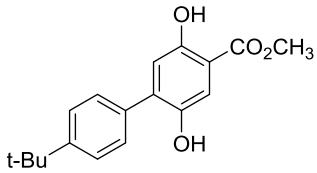
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.2, 155.2, 145.1, 136.8, 136.5, 134.7, 130.8, 129.7, 129.1, 126.5, 118.6, 114.9, 112.1, 52.4, 19.6.



Methyl-2,5-dihydroxy-3'-methyl-[1,1'-biphenyl]-4-carboxylate

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.30 (s, 1H), 7.41 (s, 1H), 7.37 (d, *J* = 7.6 Hz, 1H), 7.29 – 7.22 (m, 3H), 6.89 (s, 1H), 4.93 (s, 1H), 3.95 (s, 3H), 2.41 (s, 3H);

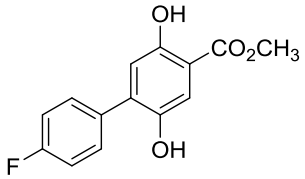
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.1, 155.3, 144.9, 139.2, 136.6, 135.8, 129.5, 129.4, 129.3, 125.7, 118.5, 115.4, 111.8, 52.4, 21.5 .



Methyl-4'-(tert-butyl)-2,5-dihydroxy-[1,1'-biphenyl]-4-carboxylate

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.31 (s, 1H), 7.52 (d, *J* = 7.6 Hz, 2H), 7.45– 7.40 (m, 3H), 6.91 (s, 1H), 4.89 (s, 1H), 3.96 (s, 3H), 1.37 (s, 9H);

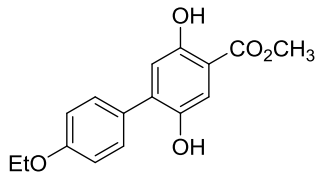
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.1, 155.4, 151.8, 144.9, 136.5, 132.9, 128.4, 126.3, 118.5, 115.4, 52.4, 34.7, 31.3.



Methyl-4'-fluoro-2,5-dihydroxy-[1,1'-biphenyl]-4-carboxylate

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.31 (s, 1H), 7.53 – 7.43 (m, 2H), 7.41 (s, 1H), 7.22 – 7.14 (m, 2H), 6.89 (s, 1H), 4.70 (s, 1H), 3.96 (d, *J* = 1.2 Hz, 3H);

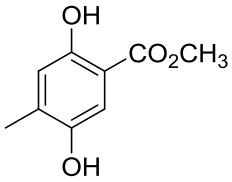
^{13}C NMR (101 MHz, $CDCl_3$) δ 169.9, 155.5, 144.8, 135.6, 132.0, 130.8, 130.7, 118.7, 116.3, 116.1, 115.7, 111.9, 52.4.



Methyl-4'-ethoxy-2,5-dihydroxy-[1,1'-biphenyl]-4-carboxylate

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.30 (s, 1H), 7.45 – 7.35 (m, 3H), 7.00 (d, *J* = 7.6 Hz, 2H), 6.88 (s, 1H), 4.84 (s, 1H), 4.08 (q, *J* = 6.8 Hz, 2H), 3.95 (s, 3H), 1.45 (t, *J* = 6.8 Hz, 3H);

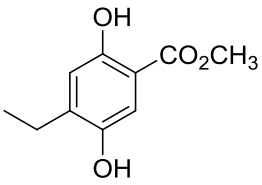
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.2, 159.3, 155.5, 144.9, 136.4, 130.0, 127.9, 118.4, 115.4, 115.3, 111.43, 63.6, 52.4, 14.8.



Methyl-2,5-dihydroxy-4-methylbenzoate

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.29 (s, 1H), 7.19 (s, 1H), 6.77 (s, 1H), 4.69 (s, 1H), 3.91 (s, 3H), 2.26 (s, 3H);

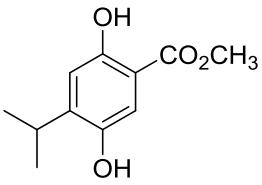
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.2, 155.7, 146.3, 134.6, 119.4, 113.9, 109.8, 52.22, 16.6.



Methyl-4-ethyl-2,5-dihydroxybenzoate

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.29 (s, 1H), 7.18 (s, 1H), 6.79 (s, 1H), 4.62 (s, 1H), 3.91 (s, 3H), 2.63 (q, *J* = 7.6 Hz, 2H), 1.23 (td, *J* = 7.6, 1.6 Hz, 3H);

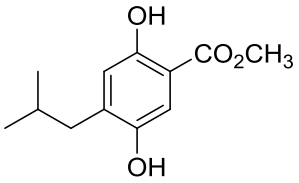
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.2, 155.9, 145.9, 140.5, 117.7, 114.2, 109.6, 52.2, 23.4, 13.3.



Methyl-2,5-dihydroxy-4-isopropylbenzoate

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.27 (s, 1H), 7.18 (s, 1H), 6.84 (s, 1H), 4.49 (s, 1H), 3.92 (s, 3H), 3.42 – 3.05 (m, 1H), 1.24 (d, *J* = 6.8 Hz, 6H);

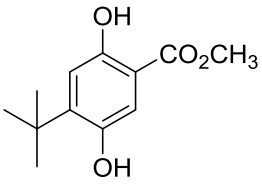
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.1, 156.3, 145.3, 145.0, 115.3, 114.3, 109.5, 52.2, 27.6, 22.2.



Methyl-2,5-dihydroxy-4-isobutylbenzoate

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.27 (s, 1H), 7.19 (s, 1H), 6.73 (s, 1H), 4.54 (s, 1H), 3.91 (s, 3H), 2.47 (d, *J* = 7.2 Hz, 2H), 2.0 –1.90 (m, 1H), 1.05 – 0.81 (m, 6H);

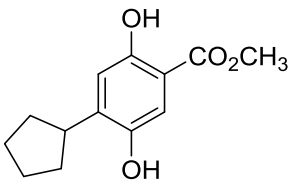
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.2, 155.6, 146.2, 138.2, 119.5, 114.4, 109.9, 52.2, 39.66, 28.6, 22.5.



Methyl-4-(tert-butyl)-2,5-dihydroxybenzoate

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.17 (s, 1H), 7.09 (s, 1H), 6.92 (s, 1H), 4.62 (s, 1H), 3.91 (s, 3H), 1.39 (s, 9H);

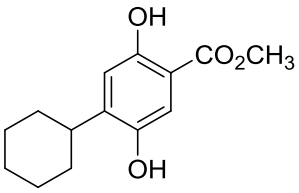
^{13}C NMR (101 MHz, $CDCl_3$) δ 169.9, 155.6, 146.7, 146.5, 116.2, 115.2, 109.5, 52.2, 35.3, 29.1.



Methyl-4-cyclopentyl-2,5-dihydroxybenzoate

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.29 (s, 1H), 7.18 (s, 1H), 6.84 (s, 1H), 4.73 (s, 1H), 3.91 (s, 3H), 3.45 – 3.06 (m, 1H), 2.10 – 2.00 (m, 2H), 1.84 – 1.74 (m, 2H), 1.75 – 1.65 (m, 2H), 1.64 – 1.52 (m, 2H);

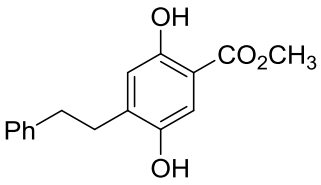
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.1, 155.9, 145.9, 142.9, 115.6, 114.2, 109.4, 52.2, 39.5, 32.6, 25.3.



Methyl-4-cyclohexyl-2,5-dihydroxybenzoate

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.29 (s, 1H), 7.18 (s, 1H), 6.82 (s, 1H), 4.75 (s, 1H), 3.90 (s, 3H), 2.90 – 2.78 (m, 1H), 1.95 – 1.74 (m, 5H), 1.47 – 1.20 (m, 5H);

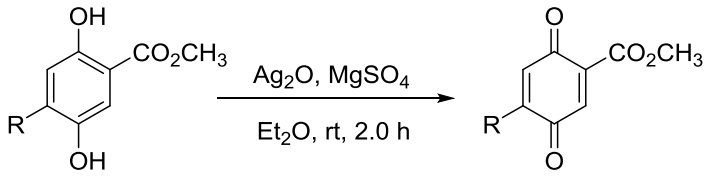
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.1, 156.0, 145.3, 144.2, 115.7, 114.3, 109.4, 52.2, 37.8, 32.6, 26.8, 26.1.



Methyl-2,5-dihydroxy-4-phenethylbenzoate

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.27 (s, 1H), 7.31 – 7.27 (t, *J* = 7.5 Hz, 2H), 7.23 – 7.19 (m, 2H), 7.18 (s, 2H), 6.76 (s, 1H), 4.38 (s, 1H), 3.91 (d, *J* = 0.6 Hz, 3H), 2.91 (s, 4H);

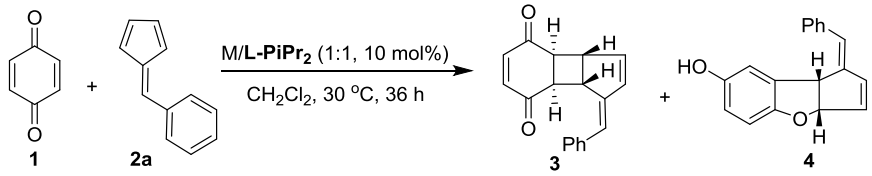
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.1, 155.9, 145.9, 141.3, 138.3, 128.5, 128.4, 126.2, 118.7, 114.7, 110.1, 52.24, 35.7, 32.7.



To a 25 mL round-bottom flask was added 2,5-dihydroxy-bznzoic acid methyl ester (1.0 mmol), $MgSO_4$ (dried at 300 ^{o}C for 2.0 h before use, 360 mg, 3 mmol), and Et_2O (dry, 20 mL). The solution was added Ag_2O (700 mg, 3.0 mmol), and then stirred for 2.0 h at room temperature. The reaction mixture was then filtered, washed with 10 mL of Et_2O (dry), and concentrated under reduced pressure at room temperature to afford the quinone product. The product was immediately used without further purification. (The product is sensitive to acid, H_2O and light, and stable at -20 ^{o}C for at least one week without any noticeable polymerization as judged by ^{1}H NMR spectroscopy)

3. Optimization of the reaction conditions

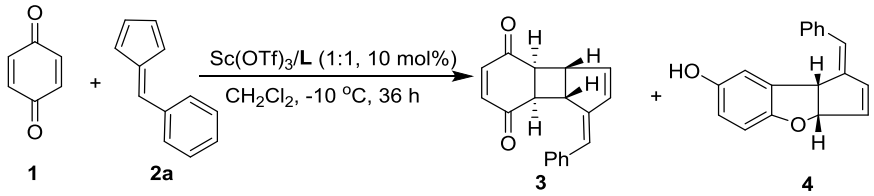
Table 1：Preliminary results of the reaction between quinone (**1**) and fulvene (**2a**)



Entry[a]	Metalsalt	3		
		Yield(%)[b]	D.r.[c]	Ee[d]
1	$Sc(OTf)_3$	83	>20:1	0
2	$Y(OTf)_3$	78	>20:1	0
3	$Yb(OTf)_3$	81	>20:1	0
4	$La(OTf)_3$	64	>20:1	0
5	$Er(OTf)_3$	67	>20:1	0

[a] Unless otherwise noted, the reactions were performed with **L-PiPr$_2$–M** (1:1, 10 mol%), **1**(0.1 mmol) and **2a** (0.15 mmol) in CH_2Cl_2 (1.0 mL) at 30 °C for 36 h. [b]Isolated yield. [c]The d.r. was determined by ^{1}H NMR and HPLC analysis. [d]Enantiometric ratio was determined by HPLCanalysis on chiral stationary phases.

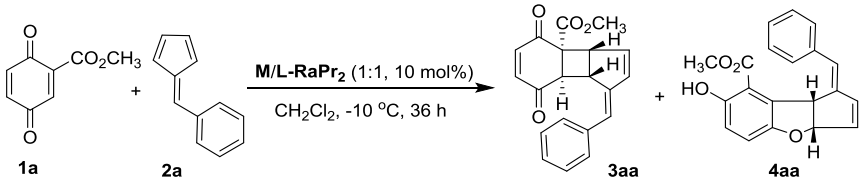
Table 2: Screening the ligands of [2+2] reaction



Entry	L	3		
		Yield(%)[b]	D.r.[c]	Ee[d]
1	**L-PiPr$_2$**	64	>20:1	11%
2	**L-PrPr$_2$**	73	>20:1	8%
3	**L-RaPr$_2$**	68	>20:1	5%

[a] Unless otherwise noted, the reactions were performed with **L–Sc(OTf)$_3$** (1:1, 10 mol%), **1**(0.1 mmol) and **2a**(0.15 mmol) in CH_2Cl_2 (1.0 mL) at −10 °C for 36 h. [b]Isolated yield. [c]The d.r. was determined by ^{1}H NMR and HPLCanalysis. [d]Enantiometric ratio was determined by HPLC analysis on chiral stationary phases.

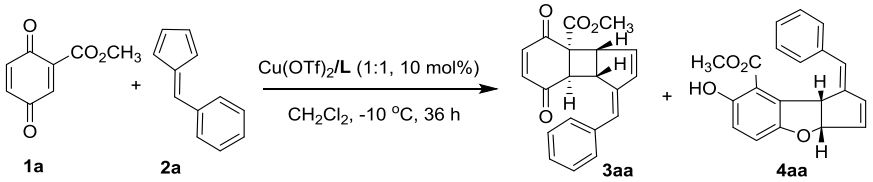
Table 3: Screening the metal salts of the reaction of quinone (**1a**)



Entry[a]	Metal salt	3aa		
		Yield(%)[b]	D.r.[c]	E.r.[d]
1	$Co(ClO_4)_2 \cdot 6H_2O$	trace	-	-
2	$Zn(OTf)_2$	25	64:36	50:50
3	$Mg(OTf)_2$	53	94:6	87:13
4	$Ni(OTf)_2$	62	73:27	72:28
5	$Cu(OTf)_2$	31	94:6	90:10

[a] Unless otherwise noted, the reactions were performed with **L-RaPr$_2$**–**M** (1:1, 10 mol%), **1a** (0.1 mmol) and **2a** (0.15 mmol) in CH_2Cl_2 (1.0 mL) at −10 °C for 36 h. [b]Isolated yield. [c]The d.r. was determined by ^{1}H NMR and HPLC analysis. [d]Enantiometric ratio of the major isomer was determined by HPLC analysis on chiral stationary phases.

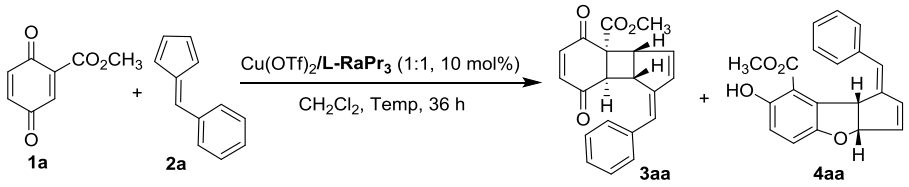
Table 4: Screening the ligands of the [2+2] reaction of quinone (**1a**)



Entry[a]	L	3aa		
		Yield(%)[b]	D.r.[c]	E.r.[d]
1	**Box**	Trace	-	-
2	**L-PiPr$_2$**	41	72:28	50:50
3	**L-PrPr$_2$**	25	95:5	94:6
4	**L-RaPr$_2$Br**	60	90:10	88:12
5	**L-RaPr$_2$^{t}Bu**	68	90:10	90:10
6	**L-RaPr$_3$**	56	94:6	94:6

[a] Unless otherwise noted, the reactions were performed with **L**–$Cu(OTf)_2$ (1:1, 10 mol%), **1a** (0.1 mmol) and **2a** (0.15 mmol) in CH_2Cl_2 (1.0 mL) at −10 °C for 36 h. [b]Isolated yield. [c]Thed.r. was determined by ^{1}H NMR and HPLC analysis. [d]Enantiometric ratio of the major isomer was determined by HPLC analysis on chiral stationary phases.

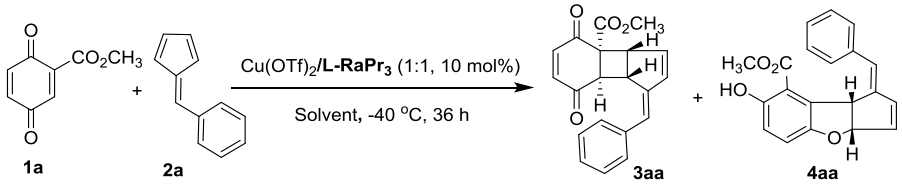
Table 5: Screening the temperature of the [2+2] reaction of quinone (**1a**)



Entry[a]	Temp.	3aa		
		Yield(%)[b]	D.r.[c]	E.r.[d]
1	−10°C	56	94:6	92:8
2	−20°C	62	94:6	94:6
3	−30°C	62	94:6	95:5
4	−40°C	63	94:6	97:3
5	−60°C	46	94:6	97:3

[a] Unless otherwise noted, the reactions were performed with **L-RaPr$_3$**–Cu(OTf)$_2$ (1:1, 10 mol%), **1a** (0.1 mmol) and **2a** (0.15 mmol) in CH_2Cl_2 (1.0 mL) at the reaction temperature for 36 h. [b]Isolated yield. [c]The d.r. was determined by ^{1}H NMR and HPLC analysis. [d]Enantiometric ratio of the major isomer was determined by HPLC analysis on chiral stationary phases.

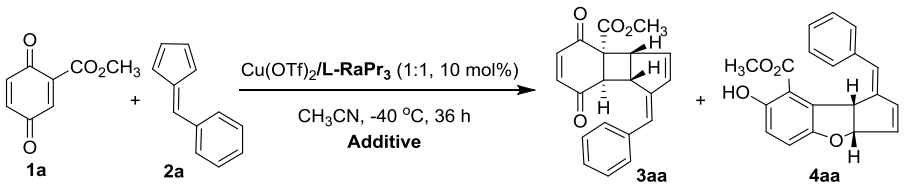
Table 6: Screening the solvents of the [2+2] reaction of quinone (**1a**)



Entry[a]	Solvent	3aa		
		Yield(%)[b]	D.r.[c]	E.r.[d]
1	$CHCl_3$	Trace	-	-
2	CH_2Cl_2	63	94:6	97:3
3	THF	Trace	-	-
4	Toluene	Trace	-	-
5	EtOAc	Trace	-	-
6	CH_3CN	73	94:6	98.5:1.5

[a] Unless otherwise noted, the reactions were performed with **L-RaPr$_3$**–Cu(OTf)$_2$ (1:1, 10 mol%), **1a** (0.1 mmol) and **2a** (0.15 mmol) in solvent (1.0 mL) at −40 °C for 36 h. [b]Isolated yield. [c]The d.r. was determined by ^{1}H NMR and HPLC analysis. [d]Enantiometric ratio of the major isomer was determined by HPLC analysis on chiral stationary phases.

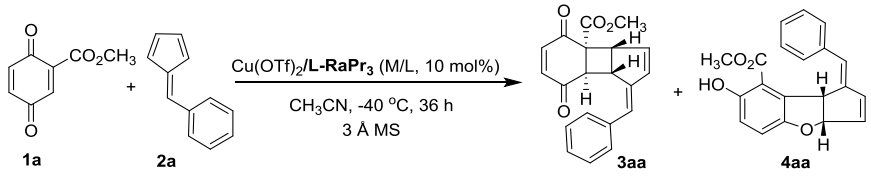
Table 7: Screening the additives of the [2+2] reaction of quinone (**1a**)



Entry[a]	Additive	3aa		
		Yield(%)[b]	D.r.[c]	E.r.[d]
1	3ÅMS	78	94:6	98.5:1.5
2	4ÅMS	74	94:6	98.5:1.5
3	5ÅMS	45	94:6	98.5:1.5

[a] Unless otherwise noted, the reactions were performed with **L-RaPr$_3$**–Cu(OTf)$_2$ (10 mol%), **1a** (0.1 mmol), MS (10 mg) and **2a** (0.15 mmol) in CH_3CN (1.0 mL) at −40 °C for 36 h. [b]Isolated yield. [c]The d.r. was determined by ^{1}H NMR and HPLC analysis. [d]Enantiometric ratio of the major isomer was determined by HPLC analysis on chiral stationary phases.

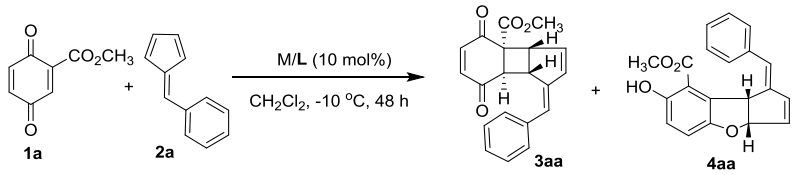
Table 8: Screening the ratio of the metal and the ligand for the [2+2] reaction



Entry[a]	M/L	3aa		
		Yield(%)[b]	D.r.[c]	E.r.[d]
1	1:1	78	94:6	98.5:1.5
2	1:1.1	82	94:6	98.5:1.5
3	1.5:1	Trace	-	-

[a] Unless otherwise noted, the reactions were performed with **L-RaPr$_3$**–Cu(OTf)$_2$ (10 mol%), **1a** (0.1 mmol), 3Å MS(10 mg) and **2a** (0.15 mmol) in CH_3CN (1.0 mL) at −40 °C for 36 h. [b]Isolated yield. [c]The d.r. was determined by ^{1}H NMR and HPLC analysis. [d]Enantiometric ratio of the major isomer was determined by HPLC analysis on chiral stationary phases.

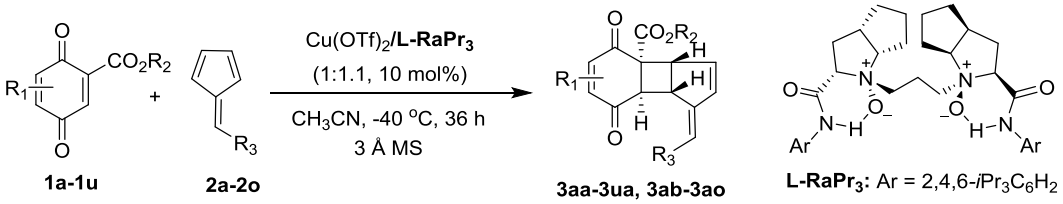
Table 9. Optimization the conditions of [3+2] reaction



Entry[a]	L	Metal	4aa		
			Yield(%)[b]	D.r. [c]	E.r. [d]
1	**L-RaPr$_2$**	In(OTf)$_3$	trace	-	-
2	**L-RaPr$_2$**	Mg(OTf)$_2$	31	N.D	56:44
3	**L-RaPr$_2$**	Mg(NTf$_2$)$_2$	45	97:3	64.5:35.5
4	**L-RaPr$_2$**	Co(ClO$_4$)$_2$.6H$_2$O	40	97:3	67:33
5	**L-RaPr$_2$**	Cu(OTf)$_2$/NaBAr$^F{}_4$	23	94:6	70.5:29.5
6	**L-RaPr$_3$**	Co(ClO$_4$)$_2$.6H$_2$O	49	99:1	56.5:43.5
7	**L-PrPr$_2$**	Co(ClO$_4$)$_2$.6H$_2$O	7	98:2	63:37
8	**L-PrPr$_3$**	Co(ClO$_4$)$_2$.6H$_2$O	8	97:3	66.5:33.5
9	**L-PiPr$_2$**	Co(ClO$_4$)$_2$.6H$_2$O	13	97:3	0
10	**L-RaPr$_3$**	Mg(NTf$_2$)$_2$	42	97:3	53:47
11	**L-PrPr$_2$**	Mg(NTf$_2$)$_2$	trace	-	-
12	**L-PrPr$_3$**	Mg(NTf$_2$)$_2$	trace	-	-
13	**L-PiPr$_2$**	Mg(NTf$_2$)$_2$	16	97:3	0

[a] Unless otherwise noted, the reactions were performed with **L**–**M** (1:1, 10 mol%), **1a** (0.1 mmol) and **2a** (0.15 mmol) in CH_2Cl_2 (1.0 mL) at −10 °C for 48 h. [b]Isolated yield. [c]The d.r. was determined by ^{1}H NMR and HPLC analysis. [d]Enantiometric ratio of the major isomer was determined by HPLC analysis on chiral stationary phases.

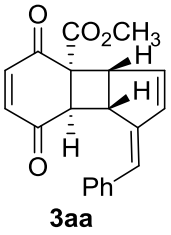
4. General procedure for the catalytic enantioselective quinone–fulvene [2 + 2] cycloadditions



In a test tube with a magnetic stirring bar *N*,*N*'-dioxide **L-RaPr$_3$** (0.011mmol), Cu(OTf)$_2$ (0.01 mmol) in CH_2Cl_2 (1.0 mL) were stirred at 35 °C for 1h. After

removing CH_2Cl_2, CH_3CN (1.0 mL) was added.The mixture was cooled to −40 °C, and the quinone (**1a-1u**) (0.1 mmol), 3Å MS (10 mg), and fulvene (**2a-2o**) (0.15 mmol, 1.5 equiv.) were added in sequence. The mixture was stirred at −40 °C for 36 h. The reaction mixture was detected by TLC. After completion, flash column chromatography was carried out to provide the desired product (**3aa–3ua, 3ab-3ao)**. The product was used immediately for HPLC and NMR analysis.

5. The analytical and spectral characterization data of the products (3aa–3ua, 3ab-3ao)



3aa

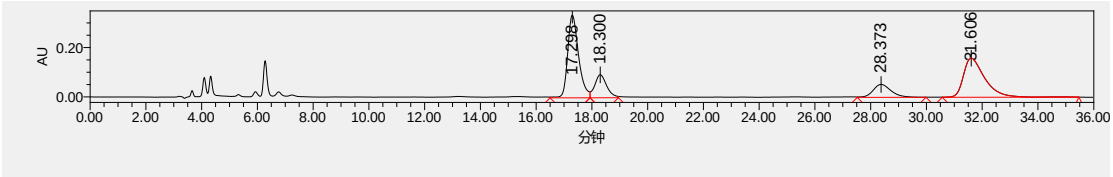
methyl-(3aR,3bR,7aR,7bS)-1-((Z)-benzylidene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3bH-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate;

yellow oil; 82% yield, 98.5:1.5 e.r., 94:6 d.r.; $[a]_D^{25}$ = −489.8 (c 0.52, CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpakIC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 17.47 min,t_2 = 18.78 min, t_3 = 28.52 min, t_4 = 31.11 min];

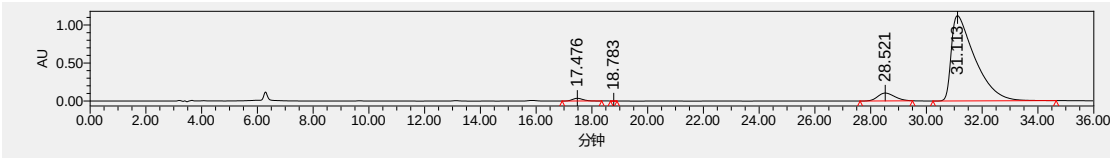
^{1}H NMR (400 MHz, CDCl3) δ 7.35 – 7.29 (m, 4H), 7.26 – 7.20 (m, 1H), 6.92 (s, 2H), 6.56 (s, 1H), 6.53 – 6.49 (m, 1H), 6.30 (dd, J = 5.2, 2.8 Hz, 1H), 3.95 – 3.89 (m, 1H), 3.84 – 3.78 (m, 2H), 3.75 (s, 3H);

^{13}C NMR (101 MHz, CDCl3) δ 195.3, 193.1, 167.0, 146.9, 141.8, 140.6, 139.9, 136.4, 133.1, 128.4, 127.9, 127.3, 125.1, 59.9, 54.0, 53.1, 52.3, 41.6;

HRMS (ESI) calcd for $[M+H]^+$, $C_{20}H_{17}O_4$, m/z: 321.1127, observed: 321.1124.



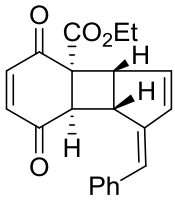
	Retention Time	Area	% Area
1	17.298	9111130	40.12
2	18.300	2778995	12.24
3	28.373	2391552	10.53
4	31.606	8427080	37.11



	Retention Time	Area	% Area
1	17.476	937704	1.32
2	18.783	852	0.00
3	28.521	4380549	6.15
4	31.113	65964609	92.54

	Retention Time	Area	% Area

1	17.476	945994	1.42
2	31.113	65751448	98.58



3ba

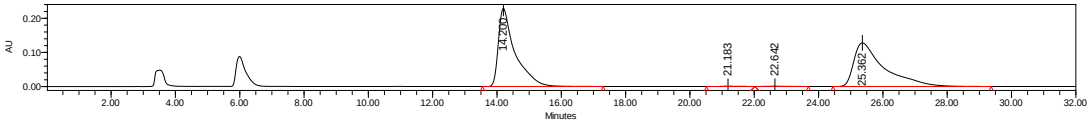
Ethyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

Yellow oil; 67% yield, 97.5:2.5 e.r., 97:3 d.r.; $[a]_D^{25}$ = −497.1 (c 0.17，CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 14.22 min,t_2 = 21.12 min, t_3 = 22.60 min, t_4 = 25.19 min];

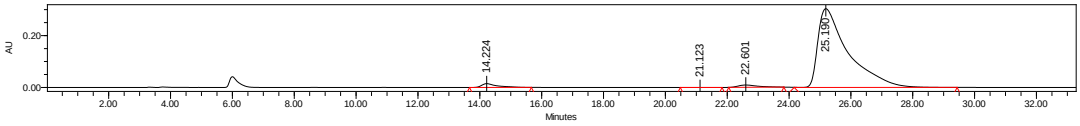
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (m, 4H), 7.22 (m, 1H), 6.91 (s, 2H), 6.55 (s, 1H), 6.50 (d, *J* = 5.4 Hz, 1H), 6.31 (dd, *J* = 5.2, 2.8 Hz, 1H), 4.29 – 4.13 (m, 2H), 3.92 –3.90 (m, 1H), 3.86 – 3.75 (m, 2H), 1.26 (t, *J* = 7.1 Hz, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.5, 193.2, 166.5, 147.1, 141.8, 140.6, 139.8, 136.4, 133.3, 128.4, 127.9, 127.3, 125.1, 123.9, 118.5, 114.7, 62.3, 59.9, 53.9, 52.4, 41.5, 14.2.

HRMS (ESI) calcd for $[M+H]^+$, $C_{21}H_{19}O_4$, m/z: 335.1283, observed: 335.1283.

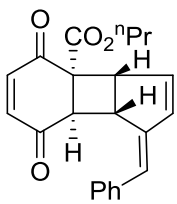


	Retention Time	Area	% Area
1	14.200	8226849	49.45
2	21.183	22193	0.13
3	22.642	31249	0.19
4	25.362	8357468	50.23



	Retention Time	Area	% Area
1	14.224	516172	2.39
2	21.123	1677	0.01
3	22.601	495254	2.29
4	25.190	20625833	95.32

	Retention Time	Area	% Area
1	14.224	516172	2.44
2	25.190	20625833	97.56

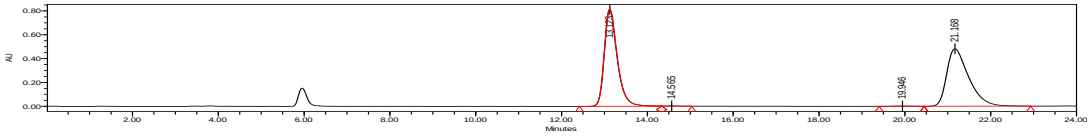


3ca

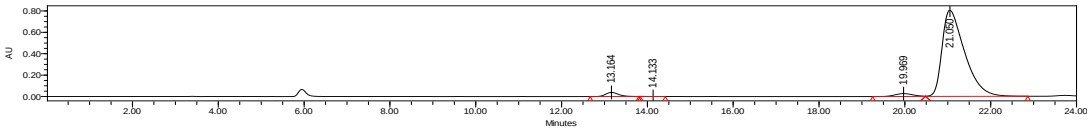
propyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

Yellow oil; 60% yield, 97:3 e.r., 97:3 d.r.; $[a]_D^{25}$ = −558.2 (c 0.69，CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpak IC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 13.16 min,t_2 = 14.13 min, t_3 = 19.96 min, t_4 = 21.05 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.27 (m, 4H), 7.25 – 7.17 (m, 1H), 6.90 (d, *J* = 1.2 Hz, 2H), 6.54 (s, 1H), 6.49 (d, *J* = 5.6 Hz, 1H), 6.34 – 6.26 (m, 1H), 4.12 – 4.06 (m, 2H), 3.92 – 3.80 (m, 1H), 3.81 – 3.76 (m, 2H), 1.69 – 1.60 (m, 2H), 0.90 (t, *J* = 7.6 3H);
^{13}C NMR (101 MHz, $CDCl_3$) δ 195.5, 193.2, 166.6, 147.1, 141.8, 140.6, 139.7, 136.4, 133.4, 128.4, 127.9, 127.3, 125.0, 67.7, 60.0, 53.8, 52.5, 41.6, 21.9, 10.3;
HRMS (ESI) calcd for $[M+H]^+$, $C_{22}H_{21}O_4$, m/z: 349.1440, observed: 349.1438.

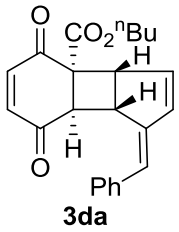


	Retention Time	Area	% Area
1	13.123	17039244	49.79
2	14.565	54167	0.16
3	19.946	65257	0.19
4	21.168	17061527	49.86



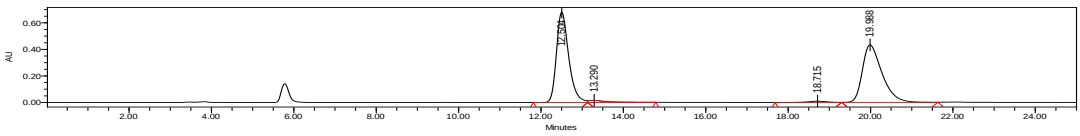
	Retention Time	Area	% Area
1	13.164	781754	2.53
2	14.133	13143	0.04
3	19.969	808541	2.61
4	21.050	29317498	94.81

	Retention Time	Area	% Area
1	13.164	763432	2.54
2	21.050	29326333	97.46

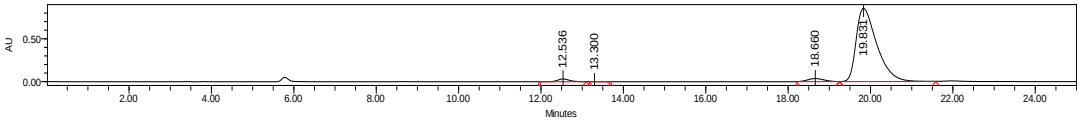


3da

butyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate
Yellow oil; 60% yield, 97:3 e.r., 97:3 d.r.; $[a]_D^{25}$ = −543.2 (c 0.83，CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 12.53 min,t_2 = 13.30 min, t_3 = 18.66 min, t_4 = 19.83 min];
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.28 (m, 4H), 7.26 – 7.17 (m, 1H), 6.91 (s, 2H), 6.55 (s, 1H), 6.49 (d, *J* = 5.6 Hz, 1H), 6.31 (d, *J* = 2.0 Hz, 1H), 4.13 (t, *J* = 6.4 Hz, 2H), 3.90 (s, 1H), 3.81 – 3.75 (m, 2H), 1.66 – 1.56 (m, 2H), 1.40 – 1.25 (m, 2H), 0.95 – 0.87 (m, 3H);
^{13}C NMR (101 MHz, $CDCl_3$) δ 195.5, 193.2, 166.6, 147.1, 141.8, 140.6, 139.7, 136.4, 133.4, 128.4, 127.9, 127.3, 125.0, 66.1, 60.0, 53.9, 52.5, 41.6, 30.5, 19.0, 13.7;
HRMS (ESI) calcd for $[M+H]^+$, $C_{23}H_{23}O_4$, m/z: 363.1596, observed: 363.1596.

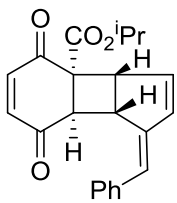


	Retention Time	Area	% Area
1	12.504	14020629	48.16
2	13.290	435973	1.50
3	18.715	285712	0.98
4	19.988	14368466	49.36



	Retention Time	Area	% Area
1	12.536	673893	2.14
2	13.300	8253	0.03
3	18.660	979105	3.11
4	19.831	29819112	94.72

	Retention Time	Area	% Area
1	12.536	673893	2.21
2	19.831	29819112	97.79



3ea

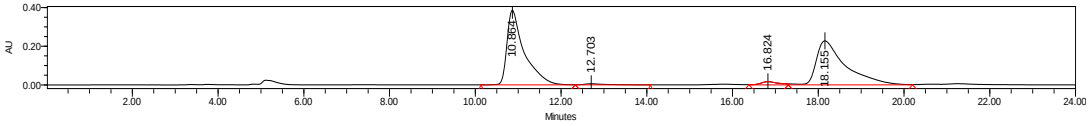
isopropyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

Yellow oil; 50% yield, 96:4 e.r., 97:3 d.r.; $[a]_D^{25}$ = −503.8 (c 0.44，CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 10.88 min, t_2 = 12.37 min, t_3 = 16.82 min, t_4 = 18.16 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.30 (m, 4H), 7.24 – 7.21 (m, 1H), 6.90 (s, 2H), 6.55 (s, 1H), 6.49 (d, *J* = 5.6 Hz, 1H), 6.31 (s, 1H), 5.10 – 4.93 (m, 1H), 3.89 (s, 1H), 3.80 – 3.75 (m, 2H), 1.23 (t, *J* = 6.8 Hz, 6H);

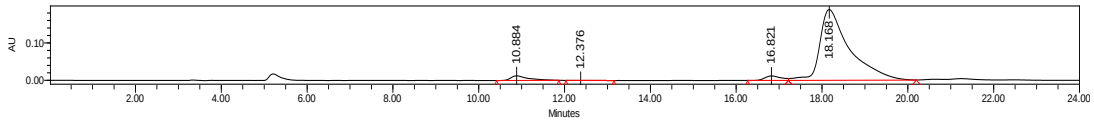
^{13}C NMR (101 MHz, $CDCl_3$) δ 195.6, 193.3, 166.1, 147.1, 141.8, 140.6, 139.6, 136.5, 133.3, 128.4, 127.9, 127.3, 124.9, 70.2, 60.1, 53.8, 52.5, 41.5, 21.8;

HRMS (ESI) calcd for $[M+H]^+$, $C_{22}H_{21}O_4$, m/z: 349.1440, observed: 349.1432.



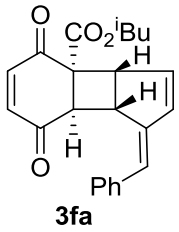
	Retention Time	Area	% Area
1	10.864	10761614	48.56
2	12.703	190802	0.86
3	16.824	463394	2.09

4	18.155	10745302	48.49



	Retention Time	Area	% Area
1	10.884	345903	3.52
2	12.376	3310	0.03
3	16.821	316750	3.23
4	18.168	9150491	93.22

	Retention Time	Area	% Area
1	10.884	345903	3.64
2	18.168	9150491	96.36



3fa

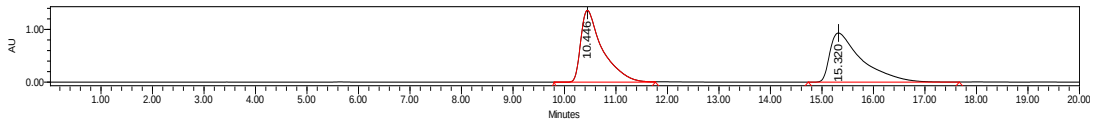
isobutyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

Yellow oil; 65% yield, 98:2 e.r., >20:1 d.r.; $[a]_D^{25}$ = −567.3 (c 0.56，CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpak IC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 10.52 min, t_2 = 15.41 min];

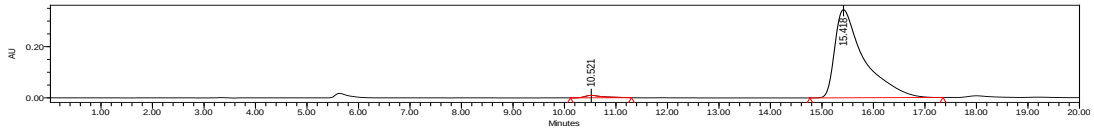
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.30 (m, 4H), 7.25 – 7.17 (m, 1H), 6.91 (s, 2H), 6.55 (s, 1H), 6.49 (d, *J* = 5.2 Hz, 1H), 6.32 (dd, *J* = 4.8, 2.4 Hz, 1H), 3.95 – 3.86 (m, 3H), 3.85 – 3.72 (m, 2H), 1.96 – 1.86 (m, 1H), 0.89 (d, *J* = 6.4 Hz, 6H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.5, 193.2, 166.6, 147.1, 141.7, 140.6, 139.7, 136.5, 133.4, 128.4, 127.9, 127.3, 125.0, 72.1, 60.0, 53.8, 52.6, 41.6, 27.7, 18.9;

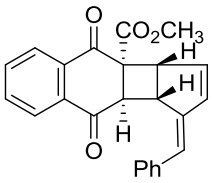
HRMS (ESI) calcd for $[M+H]^+$, $C_{23}H_{23}O$, m/z:363.1596, observed: 363.1592.



	Retention Time	Area	% Area
1	10.446	39816487	49.70
2	15.320	40292994	50.30



	Retention Time	Area	% Area
1	10.521	240060	1.74
2	15.418	13571420	98.26



3ga

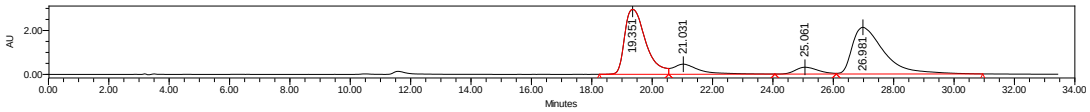
Methyl-(3aR,3bR,9aR,9bS)-1-((Z)-benzylidene)-4,9-dioxo-1,3a,4,9,9a,9b-hexahydro-3bH-cyclopenta[3,4]cyclobuta[1,2-b]naphthalene-3b-carboxylate；

Yellow oil; 55% yield, 98.5:1.5 e.r., 95:5 d.r.; $[a]_D^{25}$ = −517.3 (c 0.34, CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak Lux 5u Cellulose-2, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 19.34 min,t_2 = 20.37 min, t_3 = 24.71 min,t_4 = 26.71 min];

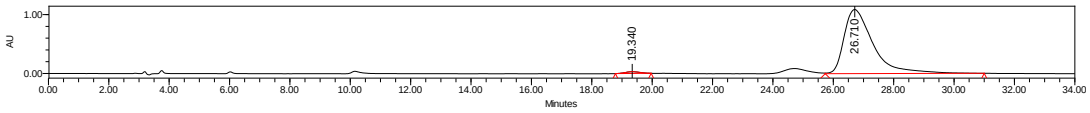
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (dt, *J* = 4.0, 2.1 Hz, 1H), 8.17 – 8.10 (m, 1H), 7.89 – 7.76 (m, 2H), 7.34 – 7.25 (m, 4H), 7.22 – 7.13 (m, 1H), 6.54 (s, 1H), 6.52 – 6.48 (m, 1H), 6.37 (dd, *J* = 5.5, 2.9 Hz, 1H), 3.95 (dd, *J* = 10.2, 4.3 Hz, 2H), 3.79 (t, *J* = 6.6 Hz, 1H), 3.69 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 193.8, 192.3, 167.7, 147.4, 139.7, 136.6, 135.4,135.3, 134.6, 134.6, 133.7, 128.4,128.3, 128.0, 127.9, 127.5, 127.1, 124.8, 60.7, 54.1, 53.3, 53.0, 41.7.

HRMS (ESI) calcd for $[M+H]^+$,$C_{24}H_{19}O_4$, m/z:371.1283, observed: 371.1281;

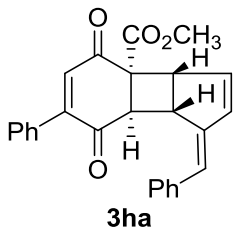


	Retention Time	Area	% Area
1	19.351	142814496	41.63
2	21.031	28049672	8.18
3	25.061	16701876	4.87
4	26.981	155522506	45.33



	Retention Time	Area	% Area
1	19.340	1013538	1.38
2	26.710	72464322	98.62

	Retention Time	Area	% Area
1	19.340	1013538	1.31
2	20.376	14170	0.02
3	24.719	3980665	5.14
4	26.710	72464322	93.54

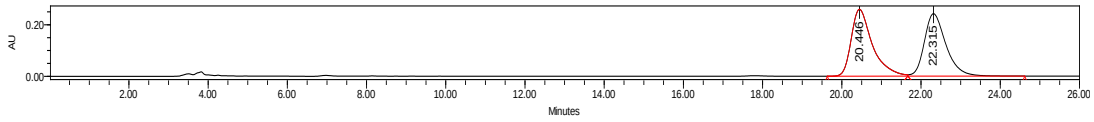


3ha

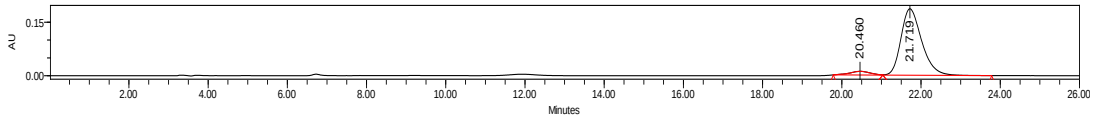
methyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-4,7-dioxo-6-phenyl-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

Yellow oil; 50% yield, 95:5 e.r.,>20:1 d.r.; $[a]_D^{25}$ = −410.7 (c 0.43，CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpakIC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 20.46 min,t_2 = 21.71 min];

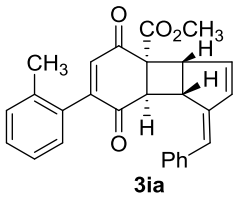
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 – 7.45 (m, 5H), 7.35 – 7.27 (m, 4H), 7.25 – 7.21 (m, 1H), 7.03 (s, 1H), 6.56 (s, 1H), 6.52 (d, *J* = 5.6 Hz, 1H), 6.41 – 6.32 (m, 1H), 3.98 (t, *J* = 6.0 Hz, 1H), 3.92 – 3.89 (m, 1H), 3.86 (d, *J* = 6.0 Hz, 1H), 3.75 (s, 3H);
^{13}C NMR (101 MHz, $CDCl_3$) δ 195.5, 192.9, 167.3, 151.3, 147.4, 139.8, 136.8, 135.6, 133.8, 132.7, 130.9, 129.1, 128.8, 128.4, 127.9, 127.2, 124.6, 60.6, 53.6, 53.0, 41.3;
HRMS (ESI) calcd for $[M+H]^+$, $C_{26}H_{21}O_4$, m/z: 397.1440, observed: 397.1441.



	Retention Time	Area	% Area
1	20.446	9647154	51.23
2	22.315	9183502	48.77



	Retention Time	Area	% Area
1	20.460	371150	5.19
2	21.719	6773829	94.81

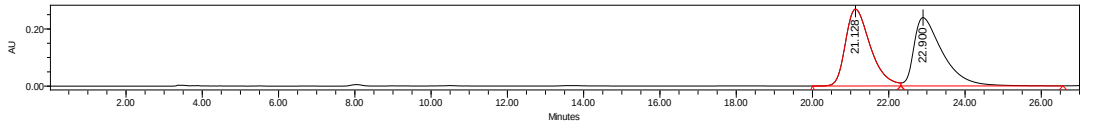


3ia

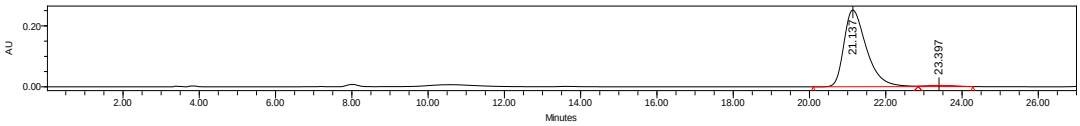
methyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-4,7-dioxo-6-(o-tolyl)-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

Yellow oil; 68% yield, 98:2 e.r., >20:1 d.r.; $[a]_D^{25}$ = −205.6 (c 1.2，CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IC, n-hexane/i-PrOH = 95/5, 1.0 mL/min, λ = 254 nm, t_1 = 21.13 min, t_2 = 23.39 min];

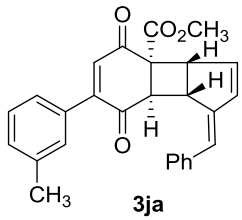
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.33 (m, 3H), 7.33 – 7.24 (m, 4H), 7.20 – 7.17 (m, 1H), 7.14 – 7.12 (m, 1H), 6.85 (s, 1H), 6.57 (s, 1H), 6.54 – 6.49 (m, 1H), 6.32 (dd, *J* = 5.6, 2.8 Hz, 1H), 4.02 – 3.98 (m, 1H), 3.94 – 3.89 (m, 2H), 3.77 (s, 3H), 2.20 (s, 3H)；
^{13}C NMR (101 MHz, $CDCl_3$) δ 194.7, 192.9, 167.3, 154.4, 147.1, 140.1, 138.6, 136.5, 135.9, 133.4, 130.5, 129.8, 128.9, 128.5, 128.3, 128.0, 127.2, 125.9, 125.1, 60.9, 53.7, 53.1, 52.6, 41.6, 20.4；
HRMS (ESI) calcd for $[M+H]^+$, $C_{27}H_{23}O_4$, m/z: 411.1596, observed: 411.1593.



	Retention Time	Area	% Area
1	21.128	12160583	49.35
2	22.900	12478810	50.65



	Retention Time	Area	% Area
1	21.137	10328933	98.29
2	23.397	179496	1.71



3ja

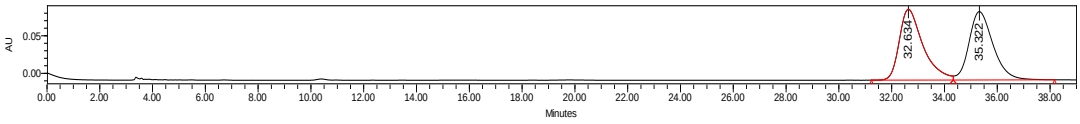
methyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-4,7-dioxo-6-(m-tolyl)-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

Yellow oil; 61% yield, 90:10 e.r., >20:1 d.r.; $[a]_D^{25}$ = −149.3 (c 1.0, CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IC, n-hexane/i-PrOH = 95/5, 1.0 mL/min, λ = 254 nm, t_1 = 33.22 min, t_2 = 34.98 min];

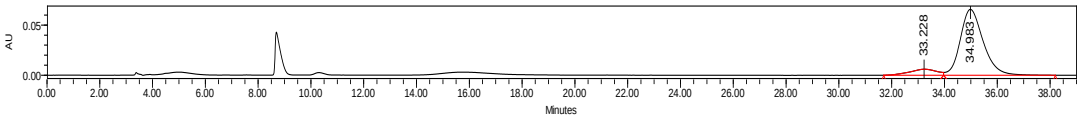
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.26 (m, 8H), 7.24 – 7.22 (m, 2H), 7.01 (d, *J* = 1.2 Hz, 1H), 6.56 (s, 1H), 6.52 (d, *J* = 5.2 Hz, 1H), 6.41 – 6.32 (m, 1H), 4.01 – 3.95 (m, 1H), 3.92 – 3.89 (m, 1H), 3.85 (d, *J* = 6.0 Hz, 1H), 3.75 (d, *J* = 2.0 Hz, 3H), 2.43 (s, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.5, 192.8, 167.4, 151.6, 147.5, 139.8, 138.4, 136.8, 135.5, 133.8, 132.7, 131.7, 129.7, 128.7, 128.5, 128.2, 127.9, 127.2, 126.2, 124.6, 60.7, 53.6, 52.9, 41.2, 21.5;

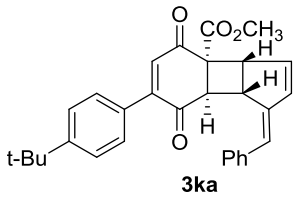
HRMS (ESI) calcd for $[M+H]^+$, $C_{27}H_{23}O_4$, m/z: 411.1596, observed: 411.1600.



	Retention Time	Area	% Area
1	32.634	5881272	50.56
2	35.322	5750123	49.44



	Retention Time	Area	% Area
1	33.228	426787	9.73
2	34.983	3959123	90.27

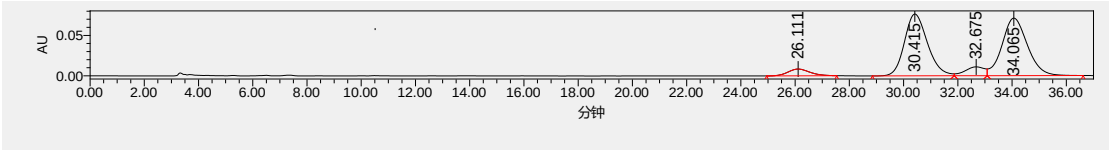


3ka

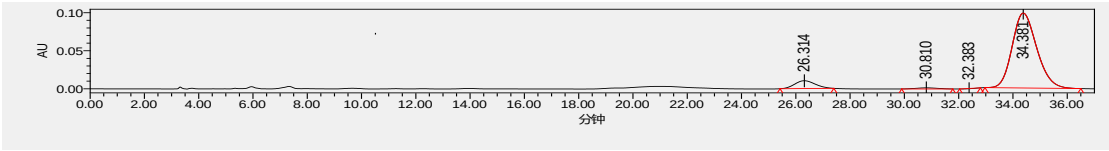
methyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-6-(4-(tert-butyl)phenyl)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

Yellow oil; 60% yield, 98:2 e.r., 92:8 d.r., $[a]_D^{25}$ = −141.1 (c 1.2, CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IC, n-hexane/i-PrOH = 95/5, 1.0 mL/min, λ = 254 nm, t_1 = 26.31 min, t_2 = 30.81 min, t_3 = 32.38 min, t_4 = 34.38 min];

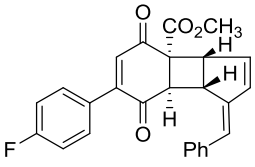
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.47 (m, 4H), 7.35 – 7.29 (m, 4H), 7.26 – 7.21 (m, 1H), 7.03 (s, 1H), 6.56 (s, 1H), 6.52 (d, *J* = 5.2 Hz, 1H), 6.37 (dd, *J* = 5.2, 2.8 Hz, 1H), 3.97 (t, *J* = 6.4 Hz, 1H), 3.89 (d, *J* = 3.6 Hz, 1H), 3.85 (d, *J* = 6.4 Hz, 1H), 3.74 (s, 3H), 1.37 (s, 9H);
^{13}C NMR (101 MHz, $CDCl_3$) δ 195.8, 192.9, 167.4, 154.7, 151.2, 147.4, 139.8, 136.7, 134.7, 133.8, 129.7, 128.9, 128.5, 128.2, 127.9, 127.2, 125.9, 124.5, 60.7, 53.6, 52.9, 41.3, 34.9, 31.2;
HRMS (ESI) calcd for $[M+H]^+$, $C_{30}H_{29}O_4$, m/z: 453.2066, observed: 453.2061.



	Retention Time	Area	% Area
1	26.111	469616	4.63
2	30.415	4512050	44.46
3	32.675	547821	5.40
4	34.065	4620052	45.52



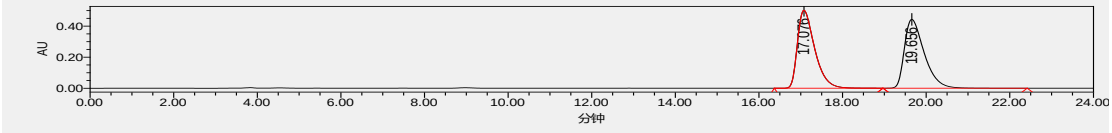
	Retention Time	Area	% Area
1	26.314	542697	7.95
2	30.810	78873	1.15
3	32.383	9900	0.14
4	34.381	6198131	90.75



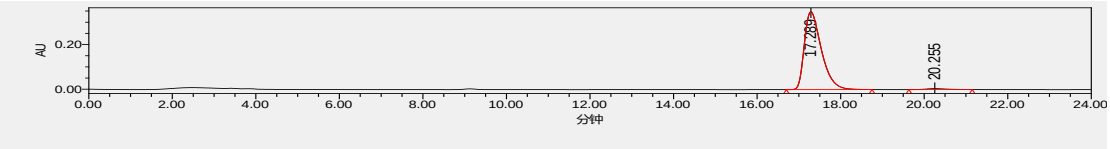
3la

Methyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-6-(4-fluorophenyl)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

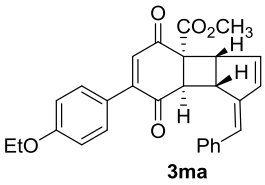
Yellow oil; 50% yield, 98.5:1.5 e.r., >20:1 d.r.; $[a]_D^{25}$ = −355.9 (c 0.72, CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IE, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 17.28 min,t_2 = 20.25 min];
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 – 7.51 (m, 2H), 7.33 – 7.31 (m, 1H), 7.30 – 7.29 (m, 2H), 7.28 (d, *J* = 1.8 Hz, 1H), 7.25 – 7.20 (m, 1H), 7.19 – 7.13 (m, 2H), 7.00 (s, 1H), 6.56 (s, 1H), 6.52 (dd, *J* = 5.2, 0.8 Hz, 1H), 6.36 (dd, *J* = 5.6, 2.8 Hz, 1H), 3.95 (t, *J* = 6.4 Hz, 1H), 3.91 – 3.87 (m, 1H), 3.85 (d, *J* = 6.4 Hz, 1H), 3.75 (s, 3H);
^{13}C NMR (101 MHz, $CDCl_3$) δ 195.3, 192.7, 167.3, 165.6, 163.1, 150.0, 147.4, 139.8, 138.1, 136.8, 135.4, 133.7, 131.4, 131.3, 128.7, 128.4, 127.9, 127.2, 124.6, 116.1, 115.9, 60.7, 53.6, 53.5, 53.0, 41.4;
HRMS (ESI) calcd for $[M+H]^+$, $C_{26}H_{20}FO_4$, m/z: 415.1346, observed: 415.1345.



	Retention Time	Area	% Area
1	17.076	14369770	49.60
2	19.656	14603878	50.40



	Retention Time	Area	% Area
1	17.289	9855230	98.59
2	20.255	141087	1.41



3ma

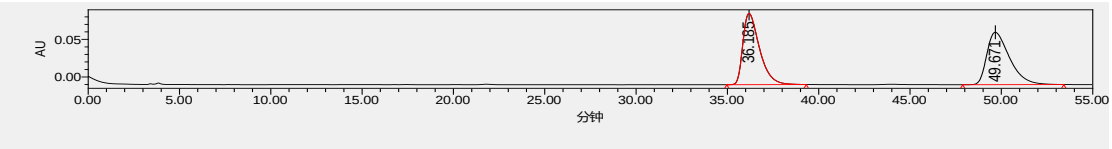
Methyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-6-(4-ethoxyphenyl)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

Yellow oil; 60% yield, 98.5:1.5 e.r., >20:1 d.r.; $[a]_D^{25} = -331.3$ (c 0.62, CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IE, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 35.70 min, t_2 = 50.15 min];

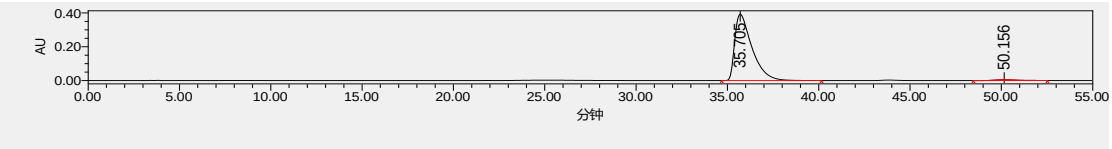
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 – 7.53 (m, 2H), 7.34 – 7.32 (m, 1H), 7.31 – 7.29 (m, 2H), 7.29 – 7,27 (m, 1H), 7.24 – 7.19 (m, 1H), 6.99 (s, 1H), 6.98 – 6.96 (m, 1H), 6.96 – 6.94 (m, 1H), 6.54 (s, 1H), 6.52 – 6.48 (m, 1H), 6.37 (dd, *J* = 5.2, 2.8 Hz, 1H), 4.11 (q, *J* = 7.2 Hz, 2H), 3.97 (t, *J* = 6.4 Hz, 1H), 3.87 – 3.85 (m, 1H), 3.81 (d, *J* = 6.4 Hz, 1H), 3.74 (s, 3H), 1.45 (t, *J* = 9.8, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.1, 192.7, 167.5, 161.5, 150.6, 147.6, 139.7, 136.8, 134.0, 133.3, 130.9, 128.5, 128.2, 127.9, 127.1, 124.5, 124.4, 114.8, 63.8, 60.6, 53.7, 53.6, 52.9, 41.2, 14.7;

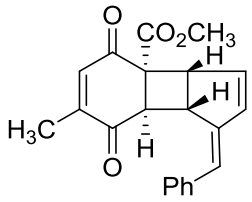
HRMS (ESI) calcd for $[M+H]^+$, $C_{28}H_{25}O_5$, m/z: 441.1702, observed: 441.1701.



	Retention Time	Area	% Area
1	36.185	5988220	49.89
2	49.671	6015235	50.11



	Retention Time	Area	% Area
1	35.705	25739380	98.53
2	50.156	384198	1.47



3na

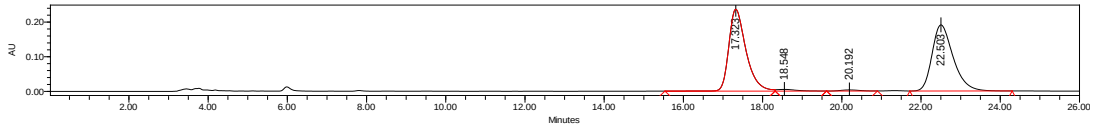
methyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-6-methyl-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

Yellow oil; 67% yield, 98:2 e.r., 97:3 d.r.; $[a]_D^{25}$ = −453.8 (c 0.60, CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 17.35 min, t_2 = 18.24 min, t_3 = 20.10 min, t_4 = 22.29 min];

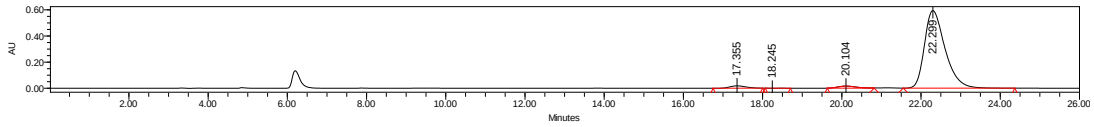
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.27 (m, 4H), 7.24 – 7.18 (M, 1H), 6.79 (s, 1H), 6.54 (s, 1H), 6.49 (d, *J* = 5.2 Hz, 1H), 6.33 – 6.28 (m, 1H), 3.85 (s, 1H), 3.75 (s, 2H), 3.73 (s, 3H), 2.15 (s, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.0, 192.6, 167.4, 152.2, 147.3, 139.7, 137.8, 136.6, 133.5, 128.3, 127.9, 127.2, 124.8, 60.5, 53.6, 52.9, 52.3, 41.8, 16.9;

HRMS (ESI) calcd for $[M+H]^+$, $C_{21}H_{19}O_4$, m/z: 335.1283, observed: 335.1287.

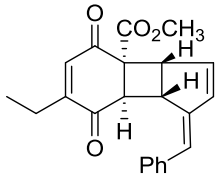


	Retention Time	Area	% Area
1	17.323	7085064	49.20
2	18.548	160482	1.11
3	20.192	105596	0.73
4	22.503	7050626	48.96



	Retention Time	Area	% Area
1	17.355	508011	2.27
2	18.245	3025	0.01
3	20.104	507825	2.27
4	22.299	21361145	95.45

	Retention Time	Area	% Area
1	17.355	508011	2.32
2	22.299	21361145	97.68

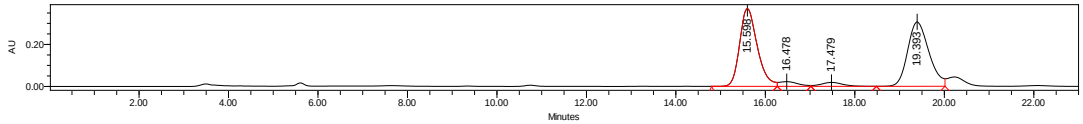


3oa

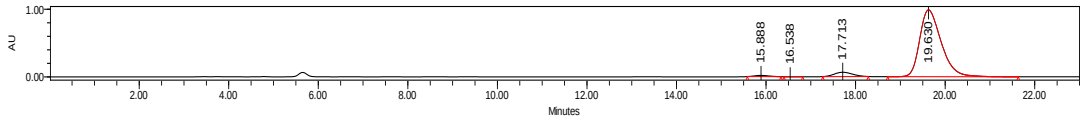
Methyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-6-ethyl-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

Yellow oil; 66% yield, 98:2 e.r., 95:5 d.r.; $[a]_D^{25}$ = −572.1 (c 0.42, CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 15.88 min, t_2 = 16.53 min, t_3 = 17.71 min,t_4 = 19.63 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ7.34 – 7.26 (m, 4H), 7.23 – 7.18 (m, 1H), 6.73 (d, *J* = 1.2 Hz, 1H), 6.54 (s, 1H), 6.48 (d, *J* = 5.2 Hz, 1H), 6.32 (dd, *J* = 5.6, 2.8 Hz, 1H), 3.87 – 3.80 (m, 1H), 3.79 – 3.74 (m, 2H), 3.73 (s, 3H), 2.67 – 2.43 (m, 2H), 1.19 (t, *J* = 7.6 Hz, 3H);
^{13}C NMR (101 MHz, $CDCl_3$) δ 195.7, 192.9, 167.4, 157.1, 147.3, 139.6, 136.6, 136.0, 133.6, 128.4, 127.9, 127.2, 124.8, 60.3, 53.6, 52.9, 52.6, 41.6, 23.3, 11.6;
HRMS (ESI) calcd for $[M+H]^+$, $C_{22}H_{21}O_4$, m/z: 349.1440, observed: 349.1441.

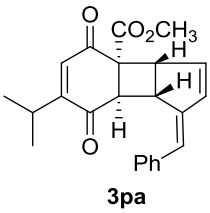


	Retention Time	Area	% Area
1	15.598	10104423	47.05
2	16.478	664397	3.09
3	17.479	635687	2.96
4	19.393	10072359	46.90



	Retention Time	Area	% Area
1	15.888	402669	1.15
2	16.538	5398	0.02
3	17.713	1793185	5.11
4	19.630	32879017	93.73

	Retention Time	Area	% Area
1	15.888	520910	1.55
2	19.630	33103195	98.45

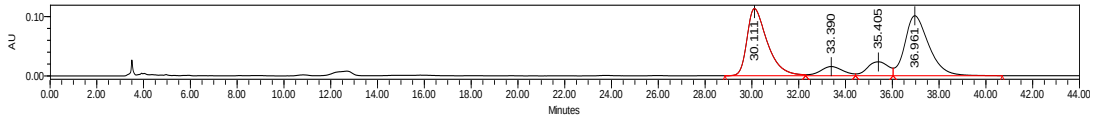


3pa

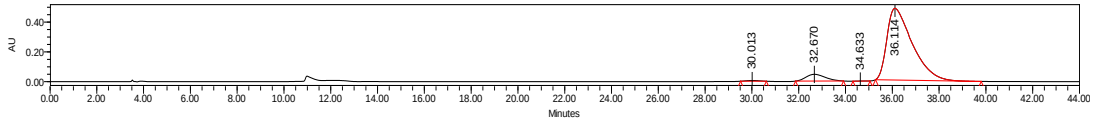
Methyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-6-isopropyl-4,7-dioxo-1, 3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

Yellow oil; 82% yield, 99:1 e.r., 93:7 d.r.; $[a]_D^{25}$ = −387.5 (c 1.2，CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IC, n-hexane/i-PrOH = 98/2, 1.0 mL/min, λ = 254 nm, t_1 = 30.01 min, t_2 = 32.67 min, t_3 = 34.63 min, t_4 = 36.11 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.25 (m, 4H), 7.23 – 7.17 (m, 1H), 6.70 (d, *J* = 1.2 Hz, 1H), 6.54 (s, 1H), 6.48 (d, *J* = 5.6 Hz, 1H), 6.32 (dd, *J* = 5.6, 2.8 Hz, 1H), 3.84 – 3.80 (m, 1H), 3.79 – 3.74 (m, 2H), 3.73 (s, 3H), 3.24 – 3.09 (m, 1H), 1.18 (dd, *J* = 6.8, 5.2 Hz, 6H);
^{13}C NMR (101 MHz, $CDCl_3$) δ 195.4, 193.2, 167.4, 161.1, 147.3, 139.7, 136.6, 134.5, 133.6, 128.4, 127.8, 127.2, 124.7, 60.1, 53.6, 52.9, 52.8 , 41.4, 27.6, 21.5, 20.4;
HRMS (ESI) calcd for $[M+H]^+$, $C_{23}H_{23}O_4$, m/z: 363.1596, observed: 363.1602.

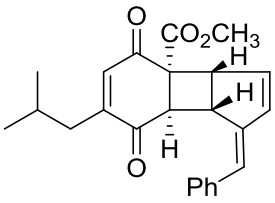


	Retention Time	Area	% Area
1	30.111	6824210	41.43
2	33.390	1037179	6.30
3	35.405	1451221	8.81
4	36.961	7160211	43.47



	Retention Time	Area	% Area
1	30.013	183245	0.47
2	32.670	2586778	6.65
3	34.633	8562	0.02
4	36.114	36114930	92.86

	Retention Time	Area	% Area
1	30.013	183245	0.50
2	36.114	36114930	99.50



3qa

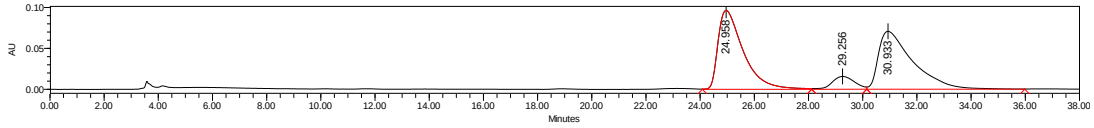
Methyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-6-isobutyl-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

Yellow oil; 83% yield, 99:1 e.r., 98:2 d.r.; $[a]_D^{25}$ = −417.1 (c 1.26, CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IE, n-hexane/i-PrOH = 98/2, 1.0 mL/min, λ = 254 nm, t_1 = 24.30 min, t_2 = 28.58 min, t_3 = 31.55 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.26 (m, 4H), 7.24 – 7.19 (m, 1H), 6.72 (s, 1H), 6.54 (s, 1H), 6.49 (dd, *J* = 5.6, 0.8 Hz, 1H), 6.33 (dd, *J* = 5.6, 2.8 Hz, 1H), 3.89 – 3.84 (m, 1H), 3.78 – 3.74 (m, 2H), 3.73 (s, 3H), 2.60 – 2.47 (m, 1H), 2.34 – 2.18 (m, 1H), 2.02 – 1.85 (m, 1H), 1.01 (d, *J* = 6.8 Hz, 3H), 0.95 (d, *J* = 6.8 Hz, 3H);

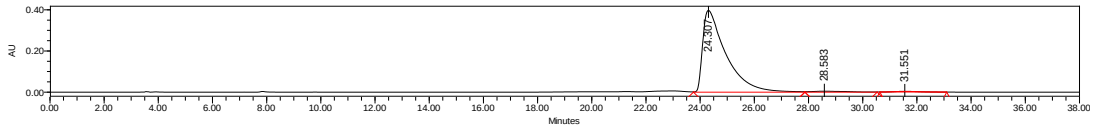
^{13}C NMR (101 MHz, $CDCl_3$) δ 195.7, 192.8, 167.4, 154.9, 147.3, 139.6, 137.7, 136.6, 133.6, 128.3, 127.8, 127.2, 124.8, 60.4, 53.6, 52.9, 52.6, 41.7, 39.3, 27.6, 22.8, 22.4;

HRMS (ESI) calcd for $[M+H]^+$, $C_{24}H_{25}O_4$, m/z: 377.1753, observed: 377.1753.



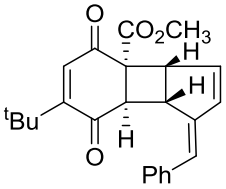
	Retention Time	Area	% Area
1	24.958	6235113	46.52

2	29.256	904569	6.75
3	30.933	6262768	46.73



	Retention Time	Area	% Area
1	24.307	23617796	97.39
2	28.583	423677	1.75
3	31.551	208712	0.86

	Retention Time	Area	% Area
1	24.307	23617796	99.12
2	31.551	208712	0.88



3ra

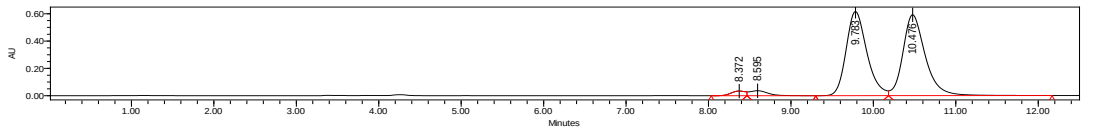
methyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-6-(tert-butyl)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

Yellow oil; 78% yield, 98:2 e.r., 95:5 d.r.; $[a]_D^{25}$ = −437.1 (c 0.87, CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 8.18 min, t_2 = 8.59 min, t_3 = 9.77 min, t_4 = 10.46 min];

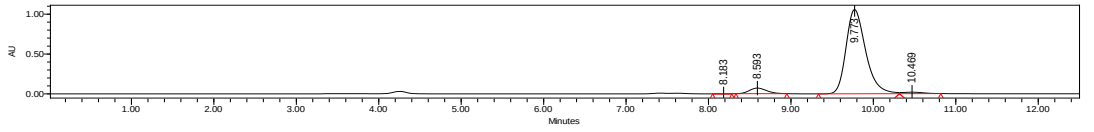
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.24 (m, 4H), 7.22 – 7.17 (m, 1H), 6.77 (s, 1H), 6.51 (s, 1H), 6.48 (d, *J* = 5.2 Hz, 1H), 6.34 – 6.28 (m, 1H), 3.82 – 3.77 (m, 2H), 3.73 (s, 3H), 3.66 (d, *J* = 5.2 Hz, 1H), 1.35 (s, 9H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.1, 193.4, 167.4, 162.8, 147.3, 140.0, 136.6, 135.4, 133.6, 128.4, 127.9, 127.1, 124.5, 60.3, 53.5, 52.9, 40.9, 36.0, 29.0;

HRMS (ESI) calcd for $[M+H]^+$, $C_{24}H_{25}O_4$, m/z: 377.1753, observed: 377.1753.



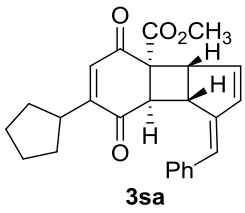
	Retention Time	Area	% Area
1	8.372	411227	1.80
2	8.595	571146	2.50
3	9.783	10748378	47.02
4	10.476	11129560	48.69



	Retention Time	Area	% Area
1	8.183	4792	0.03

2	8.593	1029104	5.47
3	9.773	17443831	92.78
4	10.469	323218	1.72

	Retention Time	Area	% Area
1	9.773	17443831	98.18
2	10.469	323218	1.82



3sa

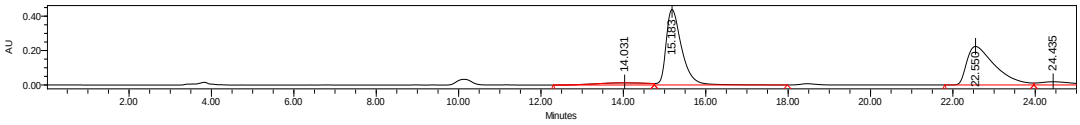
methyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-6-cyclopentyl-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

Yellow oil; 65% yield, 99:1 e.r.,98:2 d.r.; $[a]_D^{25}$ = −416.6 (c 0.38，CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IE, n-hexane/i-PrOH = 93/7, 1.0 mL/min, λ = 254 nm, t_1 = 15.03 min, t_2 = 22.88 min];

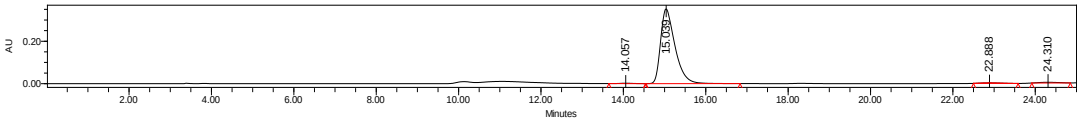
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.26 (m, 4H), 7.24 – 7.19 (m, 1H), 6.73 (d, *J* = 1.2 Hz, 1H), 6.54 (s, 1H), 6.49 (d, *J* = 5.2 Hz, 1H), 6.33 (dd, *J* = 5.6, 2.8 Hz, 1H), 3.87 – 3.81 (m, 1H), 3.7 – 3.84 (m, 2H), 3.74 (s, 3H), 3.27 – 3.14 (m, 1H), 2.21 – 2.08 (m, 1H), 2.03 – 1.91 (m, 1H), 1.86 – 1.67 (m, 4H), 1.59 – 1.48 (m, 1H), 1.49 – 1.36 (m, 1H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.9, 193.2, 167.4, 159.4, 147.3, 139.7, 136.7, 134.2, 133.6, 128.4, 127.8, 127.2, 124.7, 60.1, 53.7, 53.5, 52.9, 52.7, 41.4, 39.6, 32.4, 31.1, 25.3, 25.3;

HRMS (ESI) calcd for $[M+H]^+$, $C_{25}H_{25}O_4$, m/z: 389.1753, observed: 389.1751.

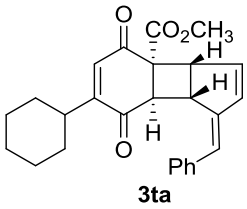


	Retention Time	Area	% Area
1	14.031	1061949	4.38
2	15.183	11535932	47.54
3	22.550	10652084	43.90
4	24.435	1016938	4.19



	Retention Time	Area	% Area
1	14.057	41239	0.47
2	15.039	8554049	97.44
3	22.888	81685	0.93
4	24.310	101725	1.16

	Retention Time	Area	% Area
1	15.039	8554049	99.05
2	22.888	81685	0.95



3ta

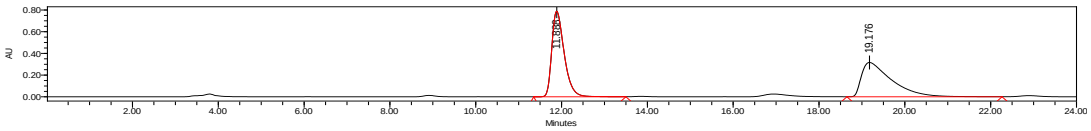
methyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-6-cyclohexyl-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

Yellow oil; 72% yield, 98.5:1.5 e.r., >20:1 d.r.; $[a]_D^{25}$ = −374.9 (c 0.72, CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IE, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 11.89 min, t_2 = 19.97 min];

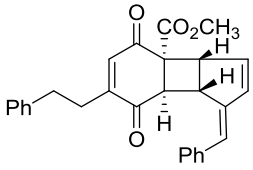
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.25 (m, 4H), 7.23 – 7.16 (m, 1H), 6.65 (d, *J* = 2.0 Hz, 1H), 6.53 (s, 1H), 6.48 (d, *J* = 5.6 Hz, 1H), 6.32 (dd, *J* = 5.6, 2.8 Hz, 1H), 3.83 – 3.81 (m, 1H), 3.76 – 3.74 (m, 1H), 3.72 (s, 3H), 2.86 – 2.80 (m, 1H), 1.96 – 1.71 (m, 6H), 1.49 – 1.39 (m, 2H), 1.31 – 1.19 (m, 2H), 1.15 – 1.05 (m, 1H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.5, 193.2, 167.4, 160.2, 147.3, 139.7, 136.6, 134.6, 133.7, 128.4, 127.9, 127.2, 124.7, 60.1, 53.6, 53.5, 52.9, 52.7, 41.4, 37.1, 32.3, 30.8, 26.4, 26.0, 25.9;

HRMS (ESI) calcd for $[M+H]^+$, $C_{26}H_{27}O_4$, m/z: 403.1909, observed: 403.1892.



	Retention Time	Area	% Area
1	11.888	15770063	50.55
2	19.176	15424035	49.45



3ua

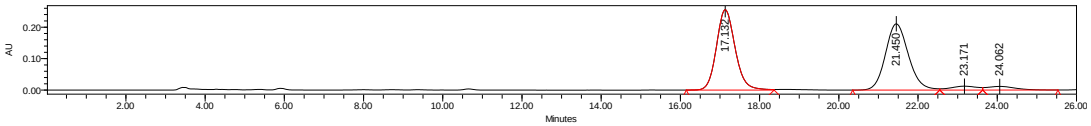
Methyl-(3a*R*,3b*R*,7a*R*,7b*S*)-1-((*Z*)-benzylidene)-4,7-dioxo-6-phenethyl-1,3a,4,7,7a,7b-hexahydro-3b*H*-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate

Yellow oil; 60% yield, 98:2 e.r., 95:5 d.r.; $[a]_D^{25}$ = −407.8 (c 1.0, CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 17.20 min, t_2 = 21.36 min, t_3 = 23.12 min, t_4 = 24.05 min];

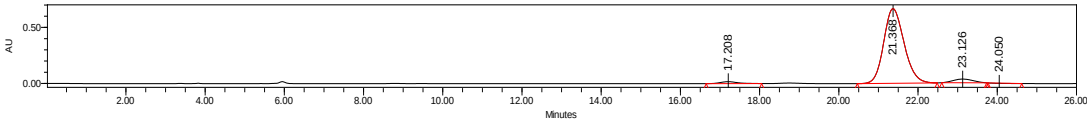
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (dd, *J* = 13.2, 4.4 Hz, 4H), 7.26 (d, *J* = 2.8 Hz, 2H), 7.23 – 7.14 (m, 4H), 6.61 (s, 1H), 6.55 (s, 1H), 6.49 (d, *J* = 5.6 Hz, 1H), 6.31 (dd, *J* = 5.2, 2.8 Hz, 1H), 3.81 (s, 1H), 3.76 – 3.74 (m, 2H), 3.72 (s, 3H), 2.93 – 2.71 (m, 4H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.6, 192.7, 167.3, 154.4, 147.3, 140.0, 139.6, 137.4, 136.6, 133.6, 128.4, 127.9, 127.3, 126.5, 124.9, 60.3, 53.1, 52.9, 52.5, 41.6, 33.8, 32.1;

HRMS (ESI) calcd for $[M+H]^+$, $C_{28}H_{25}O_4$, m/z: 425.1753, observed: 425.1748.

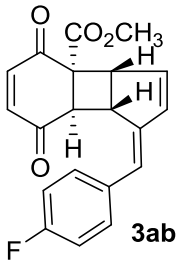


	Retention Time	Area	% Area
1	17.132	8374505	46.52
2	21.450	8480495	47.11
3	23.171	576308	3.20
4	24.062	571695	3.18



	Retention Time	Area	% Area
1	17.208	514154	2.02
2	21.368	23733165	93.22
3	23.126	1150697	4.52
4	24.050	60673	0.24

	Retention Time	Area	% Area
1	17.208	514154	2.12
2	21.368	23733165	97.88



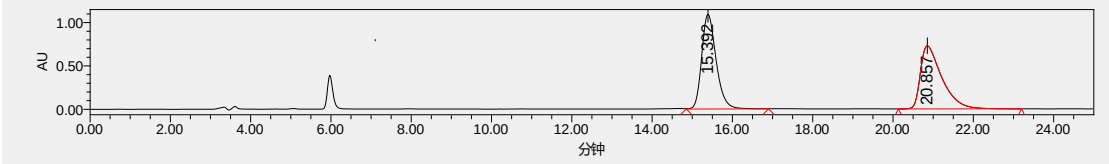
3ab

Methyl-(3aR,3bR,7aR,7bS)-1-((Z)-4-fluorobenzylidene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3bH-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate; Yellowoil; 77% yield, 98.5:1.5e.r.,>20:1 d.r.; $[a]_D^{25}$ = −519.6 (c 0.52，CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpakIC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 =15.76 min,t_2 = 20.81 min];

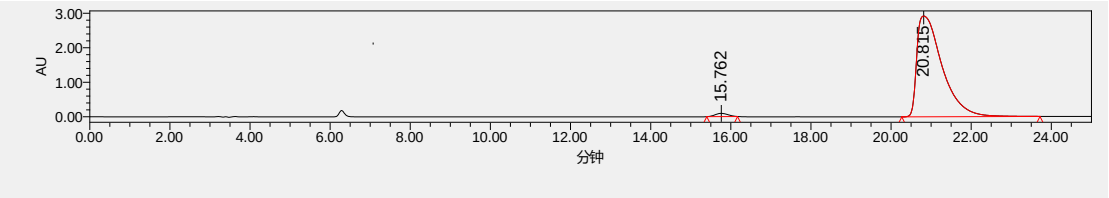
^{1}H NMR (400 MHz, CDCl3) δ 7.34 – 7.27 (m, 2H), 7.04 – 6.96 (m, 2H), 6.92 (s, 2H), 6.51 (s, 1H), 6.48 (d, J = 5.2 Hz, 1H), 6.28 (dd, J = 5.6, 2.8 Hz, 1H), 3.92-3.91 (m, 1H), 3.79 (d, J = 6.8 Hz, 1H), 3.74 (s, 3H), 3.72 (d, J = 6.8 Hz, 1H);

^{13}C NMR (101 MHz, CDCl3) δ 195.5, 193.0, 167.0, 163.2, 160.7, 146.7, 141.9, 140.7, 139.8, 133.2, 132.6, 129.6, 123.9, 115.5, 115.3, 59.9, 54.0, 53.1, 52.2, 41.4;

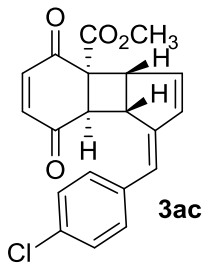
HRMS (ESI) calcd for $[M+K]^+$,$C_{20}H_{15}O_4FK$, m/z:377.0591,378.0625, observed: 377.0595,378.0647;



	Retention Time	Area	% Area
1	15.392	27321178	50.50
2	20.857	26783837	49.50



	Retention Time	Area	% Area
1	15.762	2098552	1.61
2	20.815	127868596	98.39



3ac

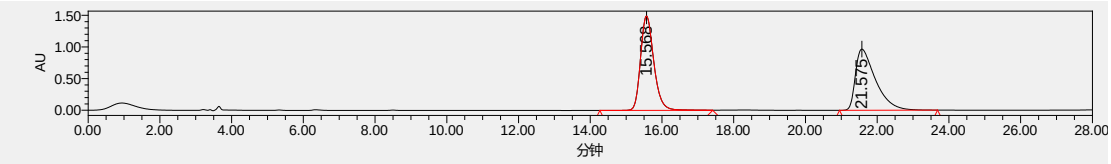
Methyl-(3aR,3bR,7aR,7bS)-1-((Z)-4-chlorobenzylidene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3bH-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate;

Yellowoil; 75% yield, 97.5:2.5e.r.,>20:1 d.r.; $[a]_D^{25}$ = −454.9 (c 0.53, CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpakIC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 =15.55 min,t_2 = 21.23 min];

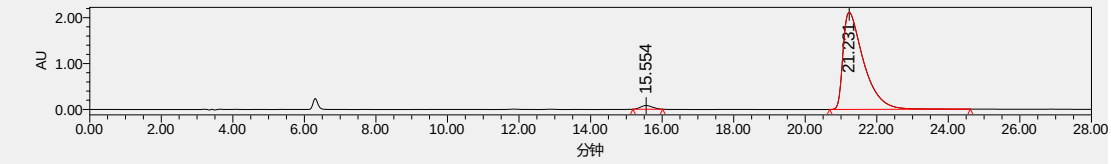
^{1}H NMR (400 MHz, CDCl3) δ 7.29 – 7.23 (m, 4H), 6.92 (s, 2H), 6.49-6.47 (m, 2H), 6.31 (dd, J = 5.6, 2.8 Hz, 1H), 3.94 – 3.91 (m, 1H), 3.78 (d, J = 6.8 Hz, 1H), 3.74 (s, 3H), 3.72 (d, J = 6.8 Hz, 1H);

^{13}C NMR (101 MHz, CDCl3) δ 195.4, 192.9, 167.0, 147.6, 141.9, 140.7, 139.8, 134.9, 133.7, 132.9, 129.2, 128.6, 123.8, 118.5, 114.7, 59.8, 54.0, 53.1, 52.0, 41.4;

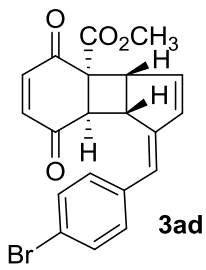
HRMS (ESI) calcd for $[M+K]^+$,$C_{20}H_{15}O_4ClK$, m/z:393.0296, 395.0266, observed: 393.0327, 395.0286;



	Retention Time	Area	% Area
1	15.568	38255600	50.53
2	21.575	37449491	49.47



	Retention Time	Area	% Area
1	15.554	1845350	2.13
2	21.231	84605647	97.87



3ad

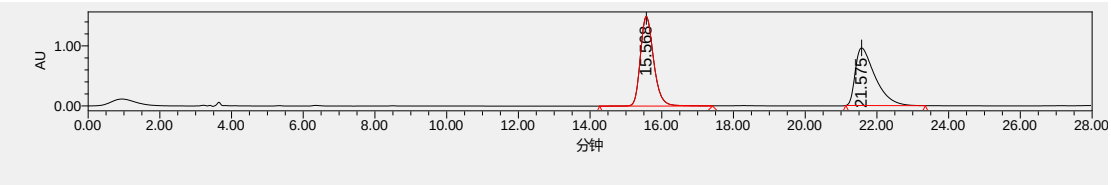
Methyl-(3aR,3bR,7aR,7bS)-1-((Z)-4-bromobenzylidene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3bH-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate;

Yellowoil; 63% yield, 97.5:2.5e.r.,>20:1 d.r.; $[a]_D^{25}$ = −389.6 (c 0.40, CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpakIC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 =16.37 min,t_2 = 22.43 min];

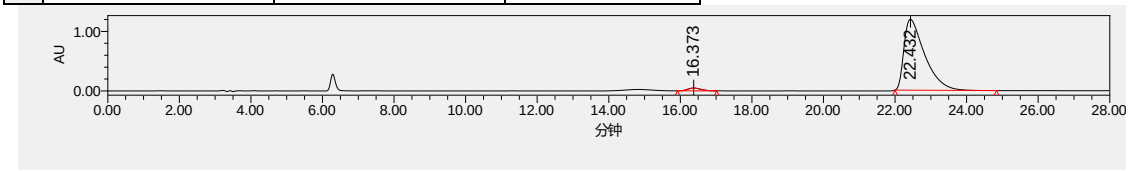
^{1}H NMR (400 MHz, CDCl3) δ 7.46 – 7.40 (m, 2H), 7.22 – 7.16 (m, 2H), 6.92 (s, 2H), 6.50 – 6.46 (m, 2H), 6.31 (dd, J = 5.6, 2.8 Hz, 1H), 3.93 – 3.90 (m, 1H), 3.78 (d, J = 6.8 Hz, 1H), 3.74 (s, 3H), 3.71 (d, J = 6.8 Hz, 1H);

^{13}C NMR (101 MHz, CDCl3) δ 195.4, 192.9, 167.0, 147.7, 141.9, 140.7, 139.8, 135.3, 133.8, 131.5, 129.5, 123.9, 121.1, 118.5, 114.7, 59.8, 54.0, 53.2, 52.0, 41.5;

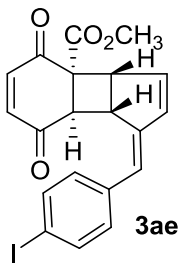
HRMS (ESI) calcd for $[M+K]^+$,$C_{20}H_{15}O_4BrK$, m/z:436.9791, 438.9770, observed: 436.9763, 438.9752;



	Retention Time	Area	% Area
1	15.568	38255600	50.97
2	21.575	36799235	49.03



	Retention Time	Area	% Area
1	16.373	1235698	2.51
2	22.432	48025369	97.49



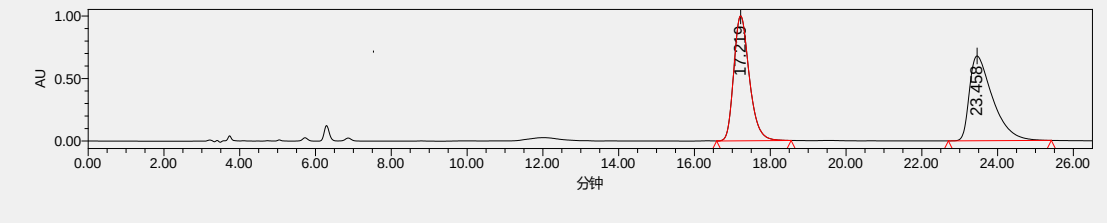
Methyl-(3aR,3bR,7aR,7bS)-1-((Z)-4-iodobenzylidene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3bH-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate;

Yellowoil; 61% yield, 97.5:2.5 e.r.,>20:1 d.r.; $[a]_D^{25}$ = −395.6 (c 0.54, CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpakIC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 =17.28 min,t_2 = 23.21 min];

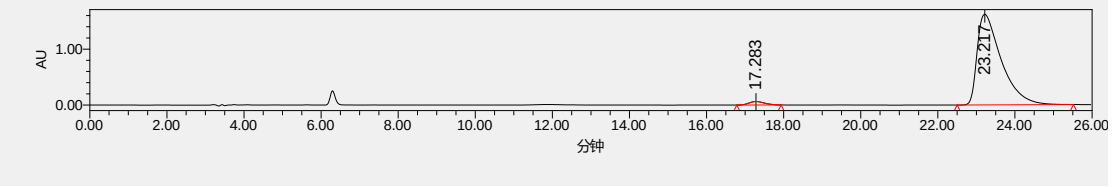
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 – 7.59 (m, 2H), 7.09 – 7.04 (m, 2H), 6.92 (s, 2H), 6.48 (d, *J* = 6.4 Hz, 1H), 6.45 (s, 1H), 6.31 (dd, *J* = 5.2, 2.8 Hz, 1H), 3.92 – 3.89 (m, 1H), 3.77 (d, *J* = 6.4 Hz, 1H), 3.74 (s, 3H), 3.71 (d, *J* = 6.4 Hz, 1H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.4, 192.9, 167.0, 147.9, 141.9, 140.7, 139.9, 137.5, 135.8, 133.9, 129.7, 124.0, 118.5, 114.7, 92.7, 59.8, 54.0, 53.2, 52.0, 41.5;

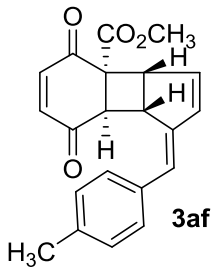
HRMS (ESI) calcd for $[M+K]^+$,$C_{20}H_{15}O_4IK$, m/z:484.9652, 485.9686, observed: 484.9659, 485.9691;



	Retention Time	Area	% Area
1	17.219	28466267	49.41
2	23.458	29143011	50.59



	Retention Time	Area	% Area
1	17.283	1737738	2.40
2	23.217	70533402	97.60



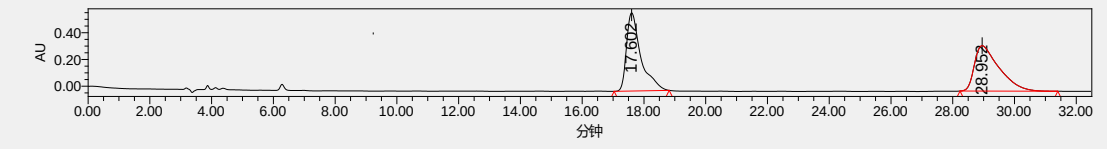
Methyl-(3aR,3bR,7aR,7bS)-1-((Z)-4-methylbenzylidene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3bH-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate;

Yellowoil; 72% yield, 98:2 e.r.,>20:1 d.r.; $[a]_D^{25}$ = −508.8 (c 0.44，CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpakIC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 =17.55 min,t_2 = 28.13 min];

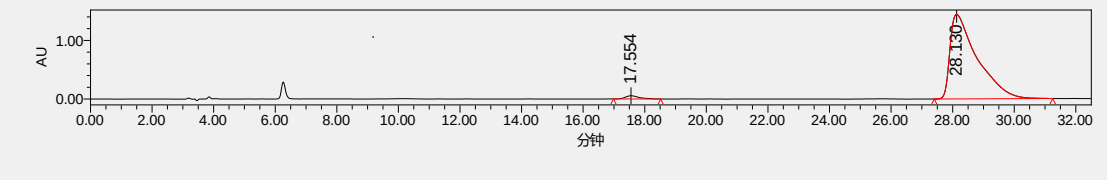
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 (d, *J* = 8.0 Hz, 2H), 7.05 (d, *J* = 8.0 Hz, 2H), 6.83 (s, 2H), 6.43 (s, 1H), 6.40 (d, *J* = 5.2 Hz, 1H), 6.18 (dd, *J* = 5.2, 2.8 Hz, 1H), 3.85 – 3.81 (m, 1H), 3.74 – 3.69 (m, 2H), 3.65 (s, 3H), 2.25 (s, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.4, 193.1, 167.1, 146.0, 141.9, 140.6, 140.1, 137.2, 133.6, 132.6, 129.2, 128.2, 127.9, 125.0, 122.2, 59.9, 54.1, 53.1, 52.4, 41.6, 21.2;

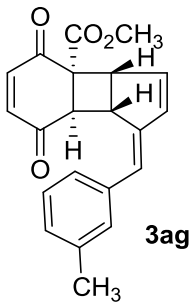
HRMS (ESI) calcd for $[M+H]^+$,$C_{21}H_{19}O_4$, m/z:335.1283, observed: 335.1283;



	Retention Time	Area	% Area
1	17.602	18879952	50.02
2	28.952	18862303	49.98



	Retention Time	Area	% Area
1	17.554	1728974	1.93
2	28.130	87986642	98.07



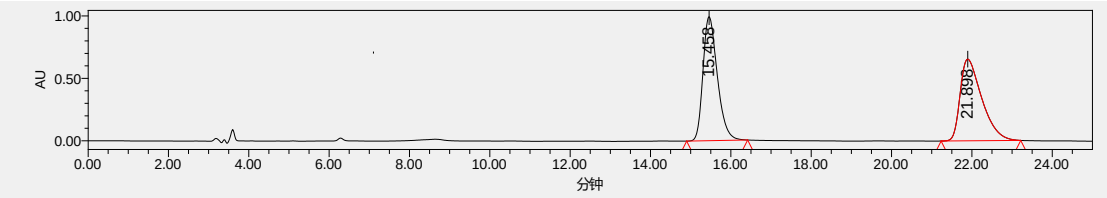
Methyl-(3aR,3bR,7aR,7bS)-1-((Z)-3-methylbenzylidene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3bH-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate;

Yellowoil; 72% yield, 98.5:1.5e.r.,>20:1 d.r.; $[a]_D^{25}$ = −375.3 (c 0.34, CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpakIC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 =15.50 min,t_2 = 21.34 min];

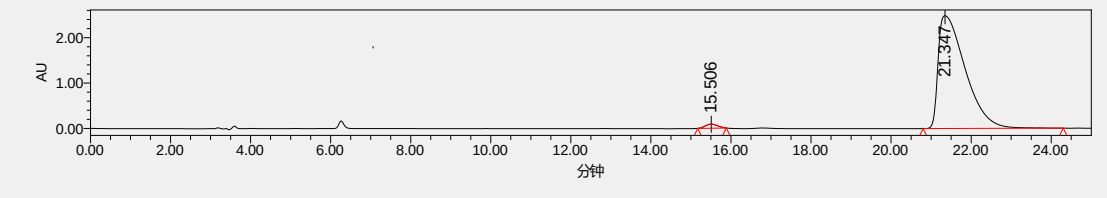
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 – 7.17 (m, 1H), 7.07 – 7.03 (m, 3H), 6.77 (d, *J* = 3.6 Hz, 2H), 6.57 (s, 1H), 6.46 (d, *J* = 5.6 Hz, 1H), 6.25 (dd, *J* = 5.6, 2.8 Hz, 1H), 3.77 (d, *J* = 4.8 Hz, 1H), 3.67 (s, 1H), 3.66 (s, 3H),3.52 (t, *J* = 6.3 Hz, 1H), 2.20 (s, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 194.8, 193.2, 167.1, 147.6, 141.8, 140.4, 139.1, 136.0, 135.6, 133.5, 129.9, 128.2, 127.4, 125.8, 123.6, 59.8, 53.5, 53.0, 52.7, 41.7, 20.0;

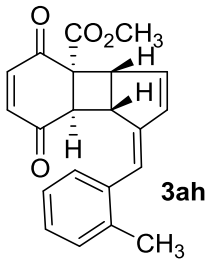
HRMS (ESI) calcd for $[M+H]^+$,$C_{21}H_{19}O_4$, m/z:335.1283, observed: 335.1283;



	Retention Time	Area	% Area
1	15.458	24681480	50.27
2	21.898	24411499	49.73



	Retention Time	Area	% Area
1	15.506	1900256	1.60
2	21.347	116697645	98.40



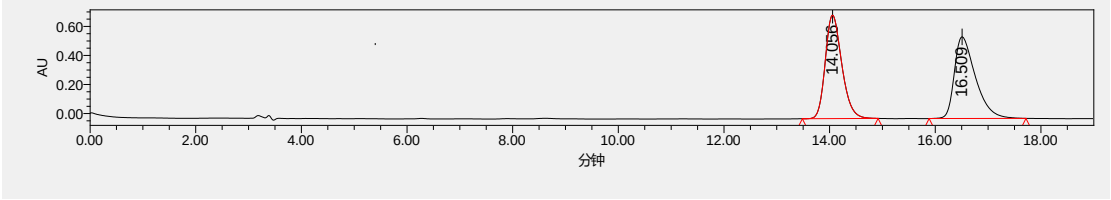
Methyl-(3aR,3bR,7aR,7bS)-1-((Z)-2-methylbenzylidene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3bH-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate;

Yellowoil; 70% yield, 97:3e.r.,>20:1 d.r.; $[a]_D^{25}$ = −527.1 (c 0.48，CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpakIC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 =14.11 min,t_2 = 16.30 min];

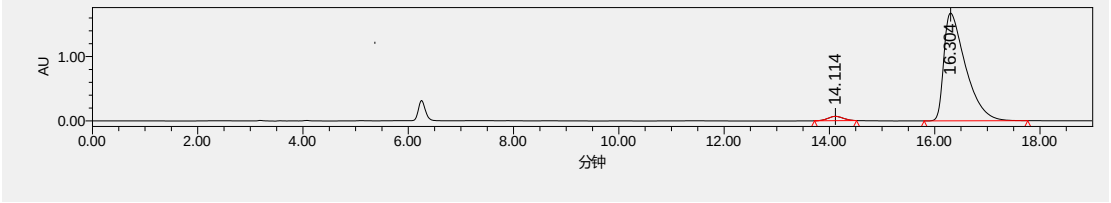
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 (t, *J* = 7.6 Hz, 1H), 7.06 (s, 1H), 7.01 (d, *J* = 7.6 Hz, 1H), 6.96 (d, *J* = 7.6 Hz, 1H), 6.83 (s, 2H), 6.44 (s, 1H), 6.41 (d, *J* = 5.6 Hz, 1H), 6.23 - 6.18 (m, 1H), 3.81 (s, 1H), 3.75 – 3.71 (m, 2H), 3.66 (s, 3H), 2.26 (s, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.3, 193.1, 167.1, 146.8, 141.9, 140.0, 138.1, 136.3, 133.0, 128.5, 128.3, 128.2, 125.3, 125.1, 59.9, 54.0, 53.1, 52.4, 41.7, 21.3;

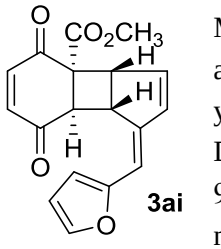
HRMS (ESI) calcd for $[M+H]^+$,$C_{21}H_{19}O_4$, m/z:335.1283, observed: 335.1282;



	Retention Time	Area	% Area
1	14.056	15483289	49.85
2	16.509	15575881	50.15



	Retention Time	Area	% Area
1	14.114	1364091	2.76
2	16.304	48032359	97.24

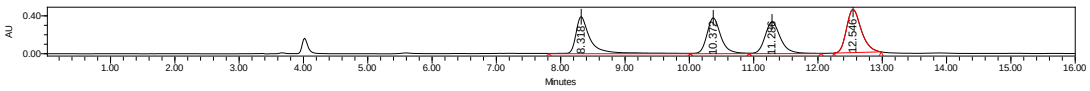


Methyl-(3aR,3bR,7aR,7bS,Z)-1-(furan-2-ylmethylene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3bH-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate；yellowoil; 61% yield, 99.5:0.5 e.r.,94:6d.r.; $[a]_D^{25}$ = −464.8 (c 0.27，CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpakIA, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 8.28 min,t_2 = 10.39 min,t_3 = 11.29 min,t_4 = 12.31 min];

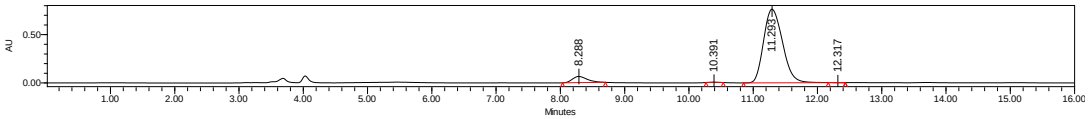
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (s, 1H), 6.92 (s, 2H), 6.47 (dd, *J* = 5.4, 1.2 Hz, 1H), 6.42 – 6.38 (m, 1H), 6.35 (dd, *J* = 5.6, 3.2 Hz, 1H), 6.29 (d, *J* = 3.6 Hz, 1H), 6.27 (s, 1H), 4.09 (t, *J* = 6.0 Hz, 1H), 3.89 – 3.82 (m, 1H), 3.72 (s, 3H), 3.52 (d, *J* = 6.4 Hz, 1H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 193.7, 193.3, 167.2, 152.3, 145.0, 142.0, 141.9, 138.6, 134.6, 111.7, 110.8, 109.9, 59.7, 53.7, 53.1, 52.9, 42.8;

HRMS (ESI) calcd for $[M+H]^+$,$C_{18}H_{15}O_5$, m/z:311.0919, observed: 311.0915;

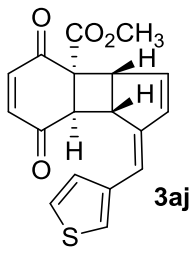


	Retention Time	Area	% Area
1	8.318	6273011	25.22
2	10.372	5676940	22.83
3	11.286	5658112	22.75
4	12.546	7260915	29.20



	Retention Time	Area	% Area
1	8.288	1031112	6.20
2	10.391	37305	0.22
3	11.293	15553016	93.56
4	12.317	1917	0.01

	Retention Time	Area	% Area
1	10.391	37305	0.24
2	11.293	15553016	99.76



3aj

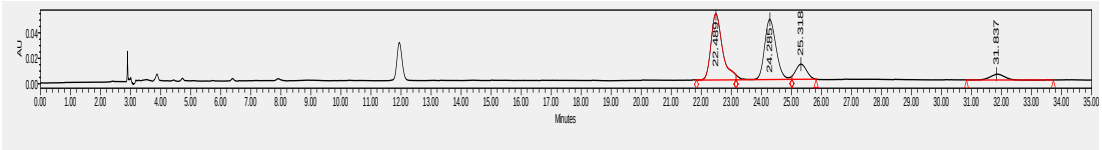
Methyl-(3aR,3bR,7aR,7bS,Z)-4,7-dioxo-1-(thiophen-3-ylmethylene)-1,3a,4,7,7a,7b-hexahydro-3bH-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate;

Yellow oil; 75% yield, 90:10 e.r., 97:3 d.r.; $[a]_D^{25} = -422.5$ (c 0.40, CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpakLux 5u cellulose-3, CO_2/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 22.89 min, t_2 = 24.13 min, t_3 = 25.35 min, t_4 = 31.76 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (s, 1H), 7.38 (s, 1H), 7.27 (s, 1H), 6.93 (s, 2H), 6.45 (d, *J* = 4.8 Hz, 1H), 6.37 (s, 1H), 6.31 (s, 1H), 6.22 (s, 1H), 3.93 (s, 2H), 3.74 (s, 3H), 3.72 – 3.65 (m, 1H);

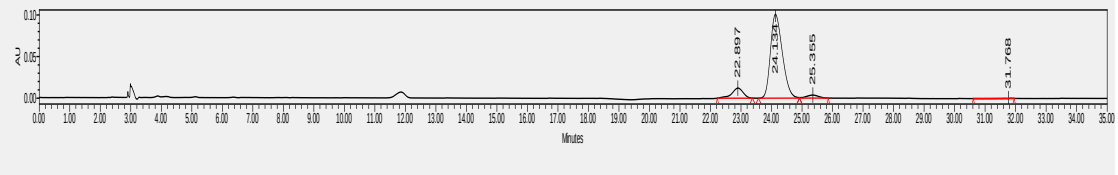
^{13}C NMR (101 MHz, $CDCl_3$) δ 195.8, 193.1, 167.0, 145.8, 143.3, 141.8, 141.3, 140.9, 139.4, 132.8, 122.3, 118.5, 114.2, 109.9, 60.1, 54.0, 53.1, 52.2, 41.6;

HRMS (ESI) calcd for $[M+H]^+$,$C_{18}H_{15}O_4S$, m/z:327.0691, observed: 327.0688;

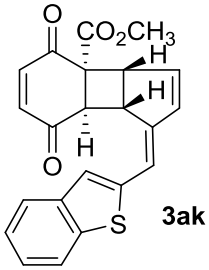


	Retention Time	Area	% Area
1	22.489	1390204	44.40
2	24.285	1285469	41.05
3	25.318	297471	9.50

4	31.837	158058	5.05



	Retention Time	Area	% Area
1	22.897	310364	9.89
2	24.134	2721932	86.75
3	25.355	97971	3.12
4	31.768	7540	0.24



3ak

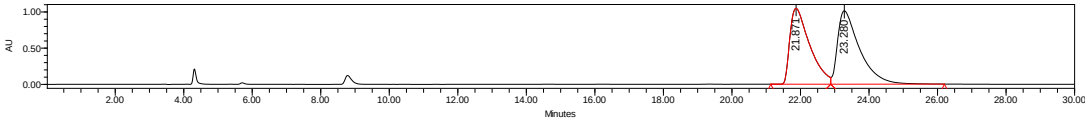
Methyl-(3aR,3bR,7aR,7bS,Z)-1-(benzo[b]thiophen-2-ylmethylene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3bH-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate;

Yellowoil; 82% yield, 98:2e.r.,>20:1d.r.; $[a]_D^{25}$ = −605.0 (c 0.56, CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpakIE, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 =22.55 min,t_2 =23.20 min];

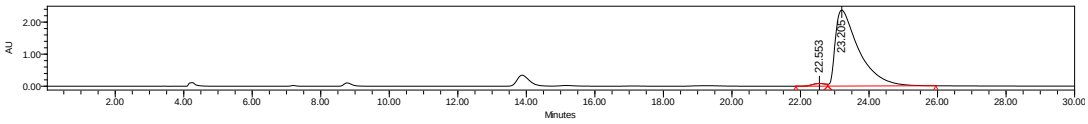
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 – 7.71 (m, 1H), 7.67 (d, *J* = 4.8 Hz, 1H), 7.58 (d, *J* = 2.8 Hz, 1H), 7.36 – 7.23 (m, 2H), 6.82 (d, *J* = 3.2 Hz, 2H), 6.71 (s, 1H), 6.52 (s, 1H), 6.26 (s, 1H), 3.83 (s, 1H), 3.79 – 3.72 (m, 1H), 3.67 (d, *J* = 3.2 Hz, 3H), 3.60 - 3.58 (m, 1H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.9, 192.9, 167.0, 148.2, 141.9, 140.9, 139.7, 139.3, 138.6, 133.9, 131.7, 124.5, 124.2, 123.3, 122.8, 121.6, 116.2, 59.9, 53.8, 53.2, 52.1, 42.2;

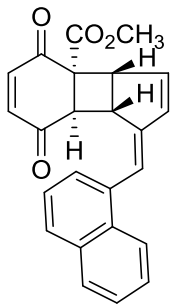
HRMS (ESI) calcd for $[M+H]^+$,$C_{22}H_{17}O_4S$, m/z:377.0848, observed: 377.0847;



	Retention Time	Area	% Area
1	21.871	42165356	48.27
2	23.280	45180457	51.73



	Retention Time	Area	% Area
1	22.553	2212595	1.93
2	23.205	112588666	98.07



3al

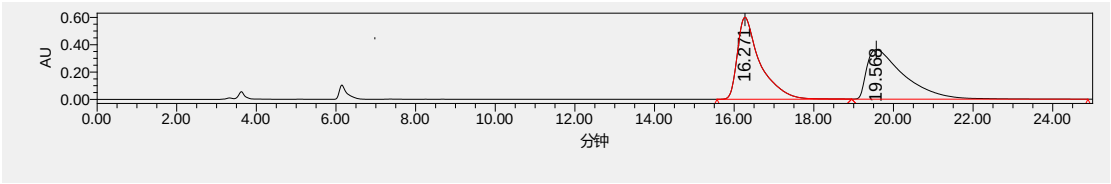
Methyl-(3aR,3bR,7aR,7bS,Z)-1-(naphthalen-1-ylmethylene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3bH-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate;

Yellowoil; 65% yield, 97:3 e.r.,>20:1 d.r.; $[a]_D^{25}$ = −225.9 (c 0.48, CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpakIC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 =16.40 min,t_2 =19.36 min];

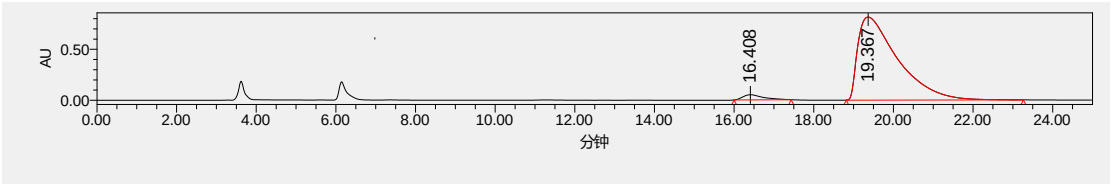
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.92 (m, 1H), 7.87 – 7.81 (m, 1H), 7.76 (d, *J* = 7.6 Hz, 1H), 7.53 – 7.42 (m, 4H), 7.15 (s, 1H), 6.86 – 6.76 (m, 2H), 6.66 (d, *J* = 5.6 Hz, 1H), 6.40 (dd, *J* = 5.6, 2.8 Hz, 1H), 3.86 – 3.81 (m, 1H), 3.80 (d, *J* = 6.8 Hz, 1H), 3.75 (s, 3H), 3.53 (t, *J* = 6.4 Hz, 1H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 194.7, 193.1, 167.1, 149.2, 141.8, 140.4, 138.7, 134.2, 133.5, 131.4, 128.5, 127.8, 126.5, 126.0, 125.8, 125.5, 124.3, 122.5, 59.8, 53.4, 53.0, 52.6, 42.1;

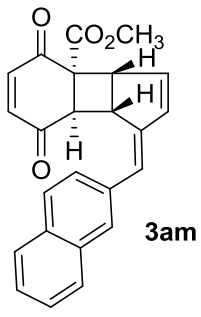
HRMS (ESI) calcd for $[M+H]^+$,$C_{24}H_{19}O_4$, m/z:371.1283, observed: 371.1283;



	Retention Time	Area	% Area
1	16.271	23718255	50.08
2	19.568	23639587	49.92



	Retention Time	Area	% Area
1	16.408	1798673	3.08
2	19.367	56632721	96.92



3am

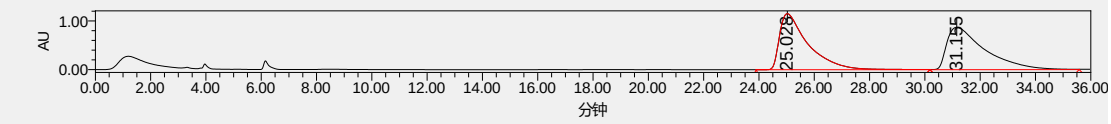
Methyl-(3aR,3bR,7aR,7bS,Z)-1-(naphthalen-2-ylmethylene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3bH-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate;

Yellowoil; 78% yield, 99:1 e.r.,>20:1 d.r.; $[a]_D^{25}$ = −392.7 (c 0.56, CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpakIC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 =25.54 min,t_2 =30.86 min];

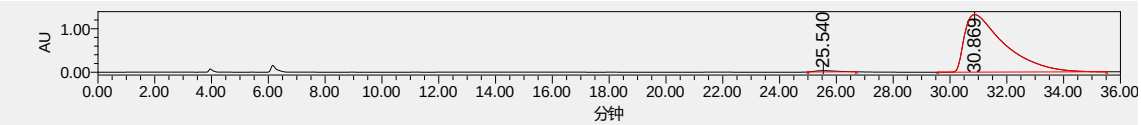
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.8 Hz, 2H), 7.75 (t, *J* = 8.0 Hz, 2H), 7.47 – 7.30 (m, 2H), 7.36 (dd, *J* = 8.4, 1.2 Hz, 1H), 6.97 – 6.86 (m, 2H), 6.69 (s, 1H), 6.53 (d, *J* = 5.2 Hz, 1H), 6.31 (dd, *J* = 5.2, 2.8 Hz, 1H), 3.91 – 3.88 (m, 1H), 3.86 (s, 1H), 3.82 (t, *J* = 6.4 Hz, 1H), 3.74 (s, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.6, 193.0, 167.1, 147.4, 141.9, 140.7, 140.0, 133.9, 133.5, 133.4, 132.6, 128.4, 127.8, 127.5, 126.6, 126.2, 125.9, 125.3, 59.9, 54.1, 53.1, 52.3, 41.8;

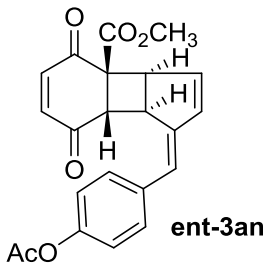
HRMS (ESI) calcd for $[M+H]^+$,$C_{24}H_{19}O_4$, m/z:371.1283, observed: 371.1288;



	Retention Time	Area	% Area
1	25.028	82232747	49.72
2	31.155	83171060	50.28



	Retention Time	Area	% Area
1	25.540	1461490	1.12
2	30.869	129336868	98.88



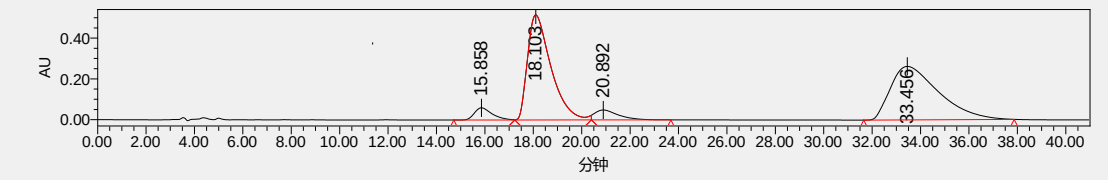
Methyl-(3aR,3bR,7aR,7bS)-1-((Z)-4-acetoxybenzylidene)-4,7-dioxo-1,3a,4,7,7a,7b-hexahydro-3bH-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate;

Yellowoil; 75% yield, 98.5:1.5 e.r.,96:4d.r.; $[a]_D^{25}$ = 456.1 (c 0.43, CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpakOD-H, n-hexane/i-PrOH = 70/30, 1.0 mL/min, λ = 254 nm,t_1=19.18 min,t_2=21.55 min,t_3 =33.22 min];

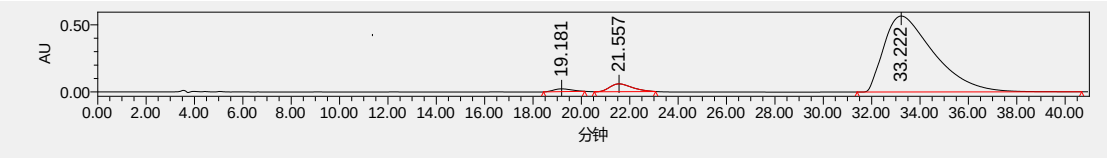
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.29 (m, 2H), 7.04 (d, *J* = 8.4 Hz, 2H), 6.91 (s, 2H), 6.53 (s, 1H), 6.49 (d, *J* = 5.2 Hz, 1H), 6.29 (dd, *J* = 5.6, 2.8 Hz, 1H), 3.94 – 3.89 (m, 1H), 3.78 (t, *J* = 6.8 Hz, 2H), 3.73 (s, 3H), 2.29 (s, 3H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 195.4, 193.0, 169.5, 167.0, 149.7, 147.1, 141.9, 140.6, 139.8, 134.2, 133.4, 129.0, 124.2, 121.5, 59.8, 54.0, 53.1, 52.2, 41.5, 21.2;

HRMS (ESI) calcd for $[M+H]^+$,$C_{22}H_{19}O_6$, m/z:379.1182, observed: 379.1179;

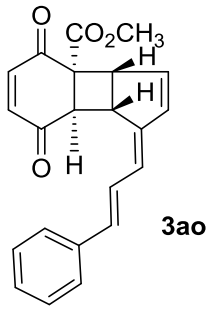


	Retention Time	Area	% Area
1	15.858	3015589	3.92
2	18.103	34585100	44.93
3	20.892	3240844	4.21
4	33.456	36139941	46.95



	Retention Time	Area	% Area
1	19.181	1086447	1.32
2	33.222	81281885	98.68

	Retention Time	Area	% Area
1	19.181	1086447	1.26
2	21.556	3677586	4.27
3	33.222	81281885	94.46



3ao

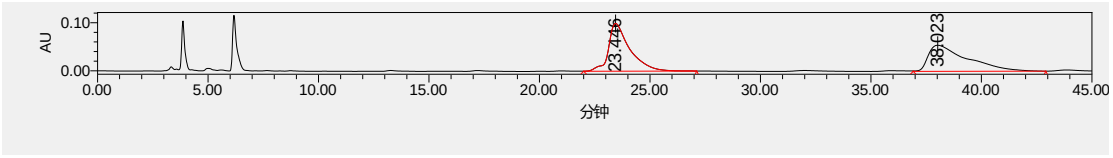
Methyl-(3aR,3bR,7aR,7bS,Z)-4,7-dioxo-1-((E)-3-phenylallylidene)-1,3a,4,7,7a,7b-hexahydro-3bH-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate;

Yellow oil; 81% yield, 97:3 e.r.,>20:1 d.r.; $[a]_D^{25}$ = −219.5 (c 0.55, CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpakIC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 23.58 min,t_2 = 37.14 min];

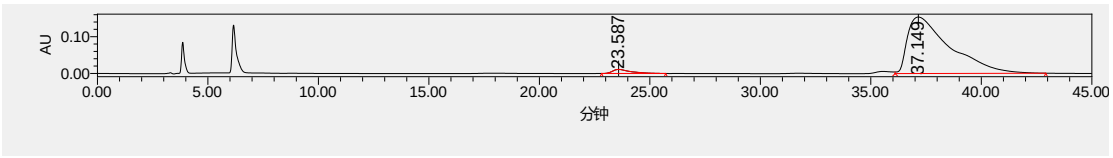
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (d, *J* = 8.0 Hz, 2H), 7.34 (t, *J* = 8.0 Hz, 2H), 7.24 – 7.20 (m, 1H), 7.08– 7.02 (m, 1H), 6.98 – 6.88 (m, 2H), 6.59 (d, *J* = 15.0 Hz, 1H), 6.47 (d, *J* = 4.8 Hz, 1H), 6.30 (d, *J* = 11.2 Hz, 1H), 6.25 (s, 1H), 3.93 (s, 2H), 3.74 (s, 3H), 3.70 – 3.67 (m, 1H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 196.0, 193.0, 167.1, 149.1, 141.9, 141.2, 138.7, 137.6, 134.6, 133.4, 128.7, 127.5, 126.6, 126.3, 124.1, 123.1, 118.5, 114.7, 61.0, 53.5, 53.2, 51.9, 41.6;

HRMS (ESI) calcd for $[M+H]^+$,$C_{22}H_{19}O_4$, m/z:347.1283, observed: 347.1286;



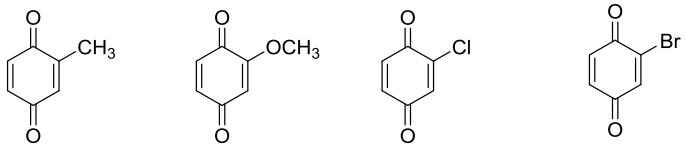
	Retention Time	Area	% Area
1	23.446	6644577	50.04
2	38.023	6633284	49.96



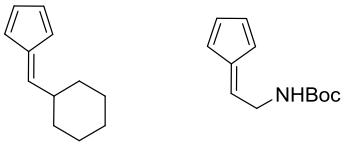
	Retention Time	Area	% Area
1	23.587	634301	3.00
2	37.149	20481712	97.00

6. Other o-substituted quinones and substituted fulvenes which are not suitable for this [2+2] cycloaddition reaction

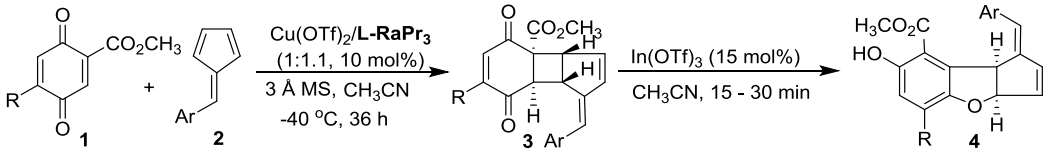
O-substituted quinones: no reaction under the optimal conditions



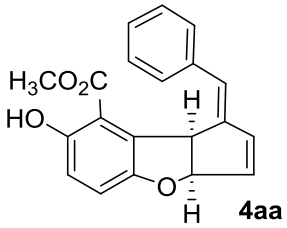
Substituted fulvenes: no reaction under the optimal conditions



7. General procedure for the isomerization of the product 3aa



In a test tube with a magnetic stirring bar, $In(OTf)_3$ (0.015 mmol)in CH_3CN (1.0 mL) wasstirred at 30 °C for 30 min. **3** (0.1 mmol)in CH_3CN (0.2 mL)was added in one-portion at −20 °C. The mixture was stirred at −20 °C for 15-30 min anddetected by TLC. After completion, flash column chromatography was carried out to provide the desired product **4**. The product was directed for HPLC and NMR analysis.



4aa

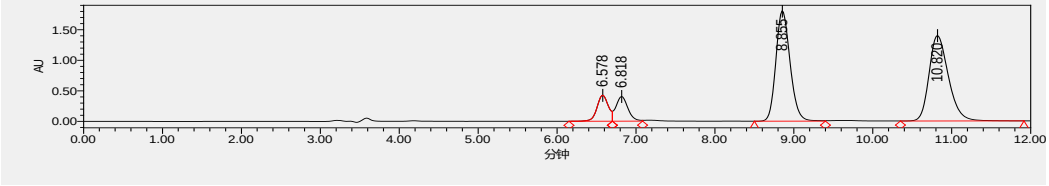
Methyl-(3aR,8bR)-1-((Z)-benzylidene)-7-hydroxy-3a,8b-dihydro-1H-cyclopenta[b]benzofuran-8-carboxylate;

Whiteoil; 95% yield, 99.5:0.5 e.r., 99:1d.r.; $[a]_D^{25}$ = −64.7 (c 0.38, CH_2Cl_2); Determined by HPLC analysis[Daicel chiralpakIA, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 6.50 min, t_2 = 8.32 min, t_3 = 10.01 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.22 (s, 1H), 7.25 – 7.18 (m, 3H), 6.95 (d, *J* = 1.6 Hz, 1H), 6.92 (d, *J* = 8.8 Hz, 2H), 6.81 (d, *J* = 8.8 Hz, 1H), 6.68 (s, 1H), 6.58 (d, *J* = 5.6 Hz, 1H), 6.20 (dd, *J* = 5.6, 2.4 Hz, 1H), 5.71 (dd, *J* = 6.4, 2.4 Hz, 1H), 4.99 (dd, *J* = 6.4, 2.0 Hz, 1H), 3.17 (s, 3H);

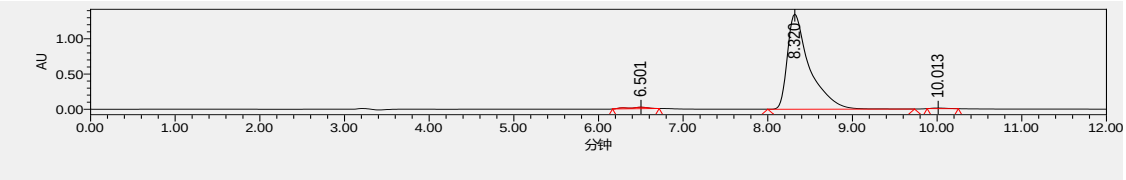
^{13}C NMR (101 MHz, $CDCl_3$) δ 169.9, 155.5, 153.4, 145.5, 143.5, 136.8, 130.8, 128.6, 128.2, 127.9, 127.0, 126.4, 117.7, 117.3, 111.8, 91.8, 51.7, 50.4;

HRMS (ESI) calcd for $[M+Na]^+$,$C_{20}H_{16}O_4Na$, m/z:343.0946, observed: 343.0947;



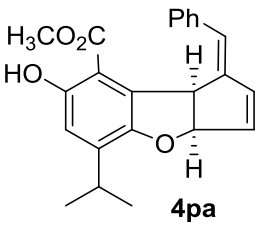
	Retention Time	Area	% Area
1	6.578	4224569	7.69
2	6.818	4269990	7.77

3	8.855	22618738	41.18
4	10.820	23807662	43.35



	Retention Time	Area	% Area
1	6.501	373648	1.50
2	8.320	24475547	98.08
3	10.013	104648	0.42

	Retention Time	Area	% Area
1	8.320	24475547	99.57
2	10.013	104648	0.43



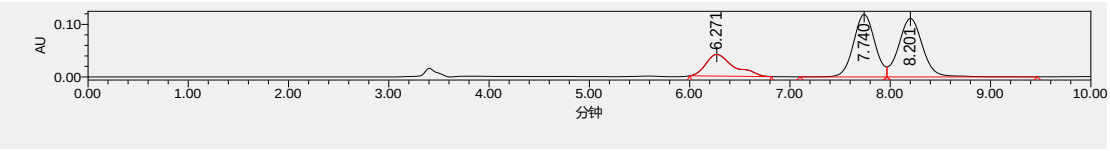
Methyl-(3a*R*,8b*R*)-1-((*Z*)-benzylidene)-7-hydroxy-5-isopropyl-3a,8b-dihydro-1*H*-cyclopenta[b]benzofuran-8-carboxylate

Colourless oil; 82% yield, 99:1e.r., 90:10 d.r.; $[a]_D^{25} = -57.9$ (c 0.56，CH_2Cl_2);Determined by HPLC analysis [Daicel chiralpak IC, n-hexane/i-PrOH = 98/2, 1.0 mL/min, λ = 254 nm, t_1 = 6.22 min, t_2 = 8.07 min];

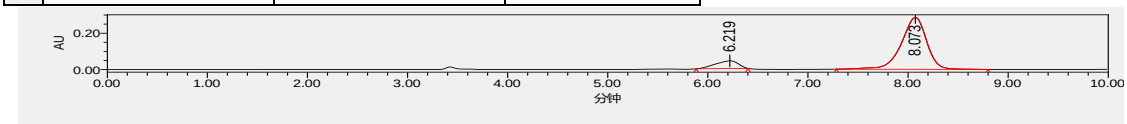
^{1}H NMR (400 MHz, $CDCl_3$) δ 10.29 (s, 1H), 7.25 – 7.16 (m, 3H), 6.98 (t, *J* = 7.2 Hz, 2H), 6.72 (s, 1H), 6.65 (s, 1H), 6.55 (d, *J* = 5.6 Hz, 1H), 6.27 – 6.10 (m, 1H), 5.70 (dd, *J* = 6.4, 1.2 Hz, 1H), 5.00 (d, *J* = 6.4 Hz, 1H), 3.11 (s, 3H), 3.05 – 3.00 (m, 1H), 1.23 (dd, *J* = 14.0, 6.8 Hz, 6H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.1, 155.9, 151.2, 145.8, 143.2, 139.7, 137.0, 131.1, 128.6, 128.1, 128.1, 127.0, 126.9, 126.1, 114.4, 109.2, 91.2, 51.5, 50.4, 28.4, 22.2, 21.6;

HRMS (ESI) calcd for $[M+H]^+$, $C_{23}H_{23}O_4$, m/z: 353.1596, observed: 353.1599.

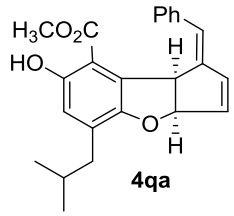


	Retention Time	Area	% Area
1	6.271	797305	18.14
2	7.740	1755614	39.95
3	8.201	1842006	41.91



	Retention Time	Area	% Area

1	6.220	590435	10.52
2	8.073	5021541	89.48



4qa

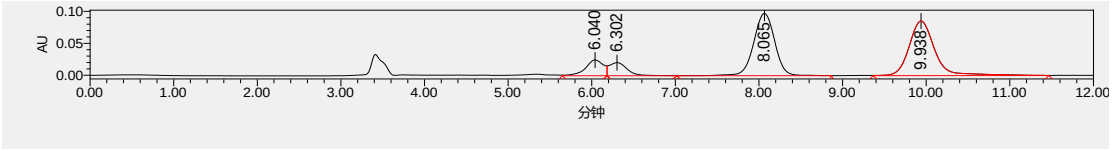
Methyl-(3a*R*,8b*R*)-1-((*Z*)-benzylidene)-7-hydroxy-5-isobutyl-3a,8b-dihydro-1*H*-cyclopenta[b]benzofuran-8-carboxylate

Colourless oil; 81% yield,97.5:2.5 e.r., 90:10 d.r.; $[a]_D^{25}$ = −70.4 (c 0.50，CH_2Cl_2);Determined by HPLC analysis [Daicel chiralpak IC, n-hexane/i-PrOH = 98/2, 1.0 mL/min, λ = 254 nm, t_1 = 6.37 min, t_2 = 8.11 min, t_3 = 10.01 min];

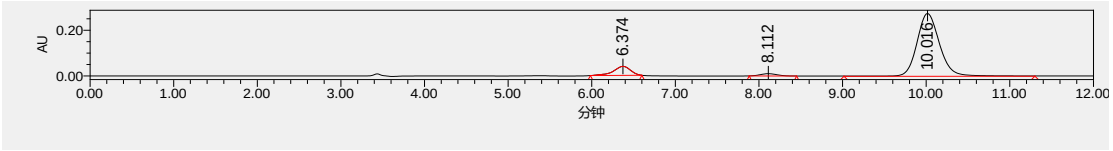
^{1}H NMR (400 MHz, $CDCl_3$) δ 10.24 (d, *J* = 1.2 Hz, 1H), 7.23 – 7.16 (m, 3H), 6.97 (d, *J* = 7.2 Hz, 2H), 6.64 (d, *J* = 8.4 Hz, 2H), 6.55 (d, *J* = 5.6 Hz, 1H), 6.24 – 6.11 (m, 1H), 5.67 (d, *J* = 6.4 Hz, 1H), 5.01 (d, *J* = 6.4 Hz, 1H), 3.12 (d, *J* = 1.2 Hz, 3H), 2.49 – 2.36 (m, 2H), 1.99 – 1.92 (m, 1H), 0.99 – 0.85 (m, 6H);

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.1, 155.4, 152.2, 145.8, 143.1, 137.0, 132.8, 131.1, 128.7, 128.1, 126.9, 126.0, 118.3, 109.4, 91.1, 51.5, 50.6, 39.5, 28.4, 22.5;

HRMS (ESI) calcd for $[M+H]^+$, $C_{24}H_{25}O_4$, m/z: 377.1753, observed: 377.1759.

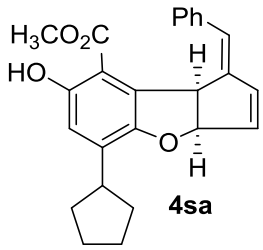


	Retention Time	Area	% Area
1	6.040	369233	8.78
2	6.302	306035	7.27
3	8.065	1712062	40.69
4	9.938	1820101	43.26



	Retention Time	Area	% Area
1	6.374	560740	9.52
2	8.112	134057	2.28
3	10.016	5194777	88.20

	Retention Time	Area	% Area
1	8.112	134057	2.52
2	10.016	5194777	97.48

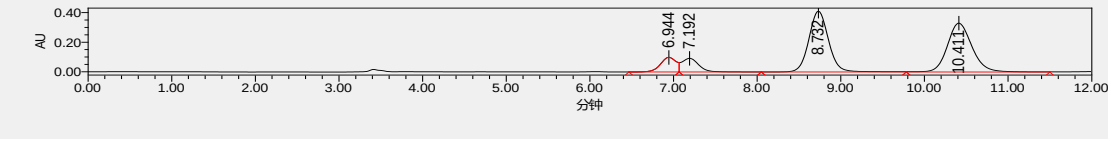


4sa

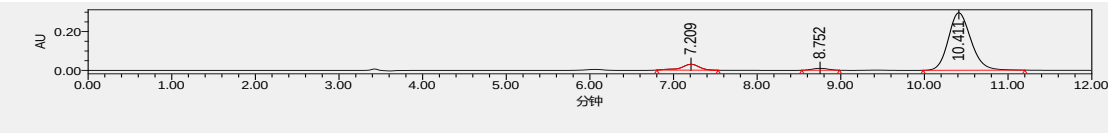
Methyl-(3a*R*,8b*R*)-1-((*Z*)-benzylidene)-5-cyclopentyl-7-hydroxy-3a,8b-dihydro-1*H*-cyclopenta[b]benzofuran-8-carboxylate

Colourless oil; 80% yield,97.5:2.5e.r., 93:7d.r.; $[a]_D^{25}$ = −106.6 (c 0.36，CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IC, n-hexane/i-PrOH = 98/2, 1.0 mL/min, λ = 254 nm, t_1 = 7.20 min, t_2 = 8.75 min, t_3 = 10.41 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.28 (s, 1H), 7.24 – 7.19 (m, 3H), 7.03 – 6.90 (m, 2H), 6.72 (s, 1H), 6.66 (s, 1H), 6.56 (d, *J* = 5.6 Hz, 1H), 6.19 (dd, *J* = 5.6, 2.4 Hz, 1H), 5.69 (dd, *J* = 6.4, 2.4 Hz, 1H), 4.99 (dd, *J* = 6.4, 1.6 Hz, 1H), 4.04 (s, 0H), 3.17 – 3.03 (m, 4H), 2.10 – 1.94 (m, 2H), 1.86 – 1.74 (m, 2H), 1.73 – 1.61 (m, 3H), 1.50 – 1.55 (m, 2H);
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.1, 155.8, 151.8, 145.8, 143.2, 137.6, 137.0, 131.1, 128.6, 128.1, 126.9, 129.9, 126.1, 115.0, 109.1, 91.2, 51.4, 50.5, 40.1, 32.9, 32.3, 25.4;
HRMS (ESI) calcd for $[M+H]^+$, $C_{25}H_{25}O_4$, m/z: 389.1753, observed: 389.1752.

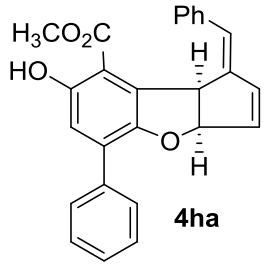


	Retention Time	Area	% Area
1	6.944	1287603	8.00
2	7.192	1307956	8.13
3	8.732	6736439	41.87
4	10.411	6756995	42.00

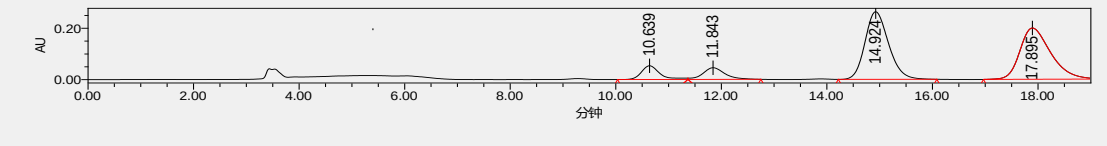


	Retention Time	Area	% Area
1	7.209	457745	7.46
2	8.752	128548	2.10
3	10.411	5547151	90.44

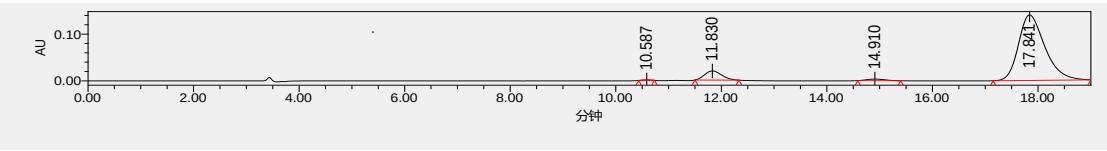
	Retention Time	Area	% Area
1	8.752	128548	2.26
2	10.411	5547151	97.74



Methyl-(3a*R*,8b*R*)-1-((*Z*)-benzylidene)-7-hydroxy-5-phenyl-3a,8b-dihydro-1*H*-cyclopenta[b]benzofuran-8-carboxylate
Colourless oil; 90% yield,98.5:1.5 e.r., 90:10 d.r.; $[a]_D^{25}$ = −166.8 (c 0.60，CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IC, n-hexane/i-PrOH = 98/2, 1.0 mL/min, λ = 254 nm, t_1 = 10.58 min, t_2 = 11.83 min, t_3 = 14.91 min, t_4 = 17.84 min];
^{1}H NMR (400 MHz, $CDCl_3$) δ 10.29 (s, 1H), 7.72 – 7.65 (m, 2H), 7.47 – 7.40 (m, 2H), 7.39 – 7.29 (m, 2H), 7.25 – 7.20 (m, 2H), 7.00 (s, 1H), 6.96 (dd, *J* = 7.2, 1.6 Hz, 2H), 6.71 (s, 1H), 6.58 (d, *J* = 5.6 Hz, 1H), 6.28 – 6.16 (m, 1H), 5.74 (dd, *J* = 6.4, 2.0 Hz, 1H), 5.03 (dd, *J* = 6.4, 2.0 Hz, 1H), 3.21 (s, 3H);
^{13}C NMR (101 MHz, $CDCl_3$) δ 169.8, 155.5, 150.9, 145.7, 143.5, 136.8, 135.9, 131.0, 129.0, 128.7, 128.6, 128.2, 128.1, 127.0, 126.5, 116.9, 110.9, 91.8, 51.6, 50.3;
HRMS (ESI) calcd for $[M+H]^+$, $C_{26}H_{21}O_4$, m/z: 397.1440, observed: 397.1440.

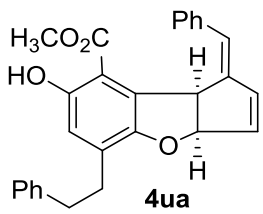


	Retention Time	Area	% Area
1	10.639	1299976	6.89
2	11.843	1312063	6.96
3	14.924	8160113	43.26
4	17.895	8092813	42.90



	Retention Time	Area	% Area
1	10.587	17141	0.31
2	11.830	497996	8.91
3	14.910	74627	1.34
4	17.841	4999181	89.45

	Retention Time	Area	% Area
1	14.910	74627	1.47
2	17.841	4999181	98.53



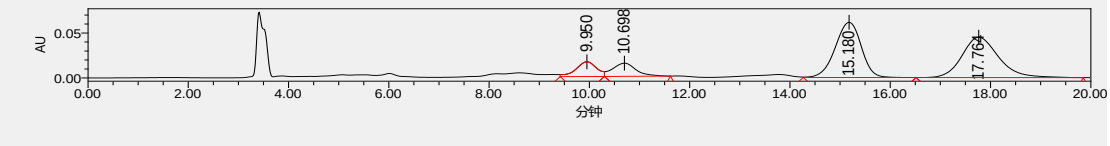
Methyl-(3a*R*,8b*R*)-1-((*Z*)-benzylidene)-7-hydroxy-5-phenethyl-3a,8b-dihydro-1*H*-cyclopenta[b]benzofuran-8-carboxylate

Colourless oil; 85% yield, 98.5:1.5 e.r., 90:10 d.r.; $[a]_D^{25}$ = −72.6 (c 0.35，CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IC, n-hexane/i-PrOH = 98/2, 1.0 mL/min, λ = 254 nm, t_1 = 9.98 min, t_2 = 10.75 min, t_3 = 15.17 min, t_4 = 17.57 min];

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.25 (d, *J* = 1.6 Hz, 1H), 7.25 – 7.14 (m, 8H), 6.93 (d, *J* = 6.8 Hz, 2H), 6.67 (s, 1H), 6.62 (s, 1H), 6.56 (d, *J* = 5.2 Hz, 1H), 6.23 – 6.11 (m, 1H), 5.67 (d, *J* = 6.4 Hz, 1H), 4.97 (d, *J* = 6.4 Hz, 1H), 3.14 (d, *J* = 1.6 Hz, 3H), 2.94 – 2.80 (m, 4H);

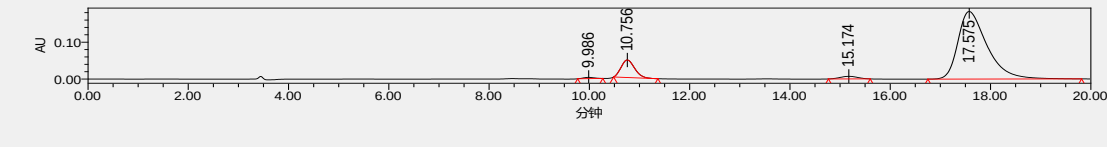
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.1, 155.6, 151.9, 145.7, 143.3, 141.4, 136.9, 132.5, 130.9, 128.5, 128.3, 128.1, 127.2, 126.9, 126.2, 125.9, 117.6, 109.6, 915, 51.5, 50.6, 35.1, 32.3;

HRMS (ESI) calcd for $[M+H]^+$, $C_{28}H_{25}O_4$, m/z: 425.1753, observed: 425.1757.



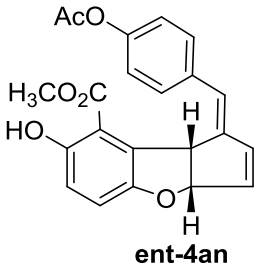
	Retention Time	Area	% Area
1	9.950	468971	8.61
2	10.698	474580	8.71
3	15.180	2220231	40.76

4	17.764	2283923	41.92



	Retention Time	Area	% Area
1	9.986	51431	0.61
2	10.756	892502	10.51
3	15.174	169494	2.00
4	17.575	7379159	86.89

	Retention Time	Area	% Area
1	15.174	188796	2.48
2	17.575	7431882	97.52



ent-4an

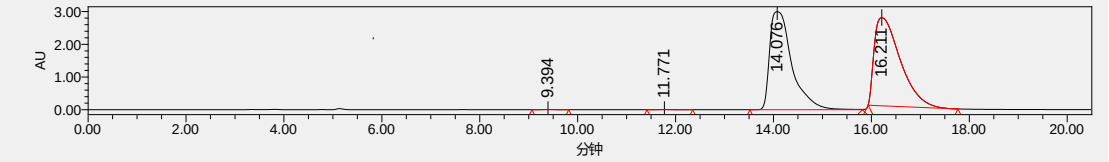
Methyl-(3aS,8bS)-1-((Z)-4-acetoxybenzylidene)-7-hydroxy-3a,8b-dihydro-1H-cyclopenta[b]benzofuran-8-carboxylate;

White oil; 95% yield, 99:1 e.r., 99:1 d.r.; $[a]_D^{25}$ = 118.9 (c 0.56, CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IE, n-hexane/i-PrOH = 80/20, 1.0 mL/min, λ = 254 nm, t_1 =14.34 min, t_2 = 16.78 min];

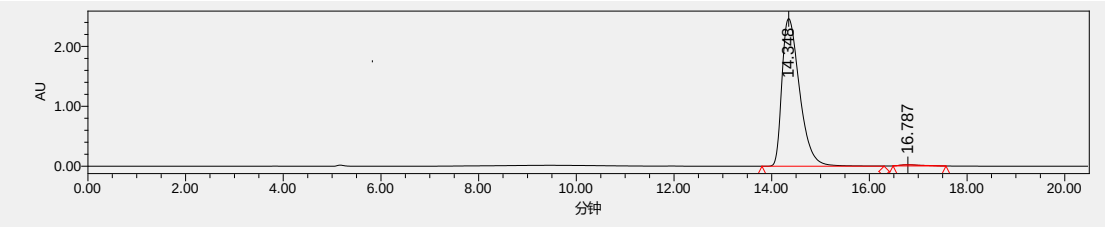
^{1}H NMR (400 MHz, $CDCl_3$) δ 10.20 (s, 1H), 6.96 (s, 4H), 6.92 (d, *J* = 8.8 Hz, 1H), 6.81 (d, *J* = 8.8 Hz, 1H), 6.63 (d, *J* = 1.2 Hz, 1H), 6.57 (d, *J* = 5.6 Hz, 1H), 6.20 (dd, *J* = 5.2, 2.0 Hz, 1H), 5.71 (dd, *J* = 6.4, 2.4 Hz, 1H), 5.00 (dd, *J* = 6.4, 2.0 Hz, 1H), 3.26 (s, 3H), 2.31 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.8, 169.5, 155.4, 153.4, 149.6, 146.0, 143.3, 134.5, 131.1, 129.6, 127.7, 125.4, 121.4, 117.8, 117.3, 111.8, 91.7, 51.8, 50.3, 21.2.

HRMS (ESI) calcd for $[M+Na]^+$,$C_{22}H_{18}O_6Na$, m/z:401.1001, observed: 401.1000;

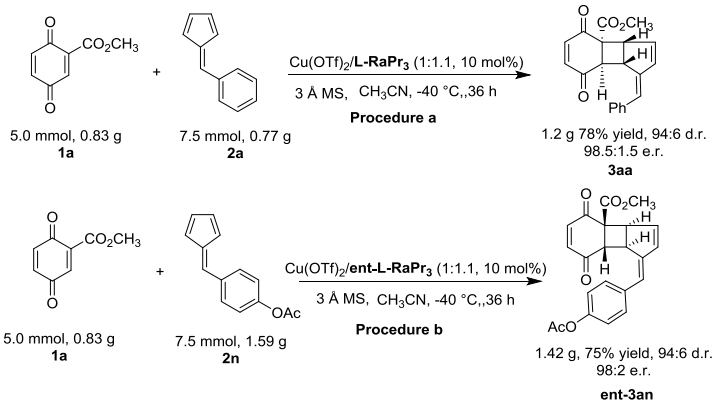


	Retention Time	Area	% Area
1	9.394	131367	0.07
2	11.771	120096	0.06
3	14.076	94533151	48.60
4	16.211	99710533	51.27



	Retention Time	Area	% Area
1	14.348	61921628	99.04
2	16.787	597984	0.96

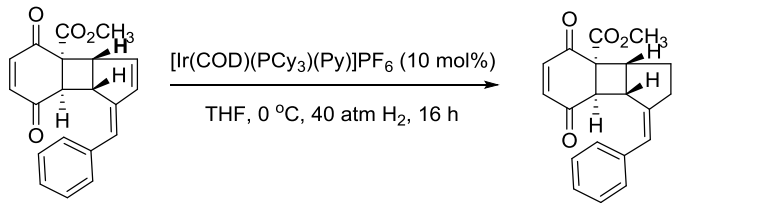
8. Gram-scale synthesis of the product 3aa and ent-3an



Procedure a: In a test tube with a magnetic stirring bar, *N*,*N*'-dioxide **L-RaPr3** (0.55 mmol), $Cu(OTf)_2$ (0.50 mmol) in CH_2Cl_2 (50 mL) were stirred at 35 °C for 1h. After removing CH_2Cl_2 and adding CH_3CN (50.0mL), quinone (**1a**) (5.0 mmol), 3Å MS (500 mg), and fulvene (**2a**) (7.5 mmol, 1.5 equiv) were added in sequence at −40 °C. The mixture was stirred at −40 °C for 36 h and detected by TLC. After completion, flash column chromatography was carried out to provide the desired product (**3aa)**. The product was used immediately for HPLC and NMR analysis.

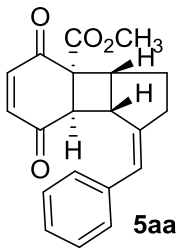
Procedure b: In a test tube with a magnetic stirring bar, *N*,*N*'-dioxide **ent-L-RaPr3**(0.55 mmol), $Cu(OTf)_2$ (0.50 mmol) in CH_2Cl_2 (50.0 mL) were stirred at 35 °C for 1h. After removing CH_2Cl_2 and adding CH_3CN (50.0 mL), quinone (**1a**) (5.0 mmol), 3Å MS (500 mg), and fulvene (**2n**) (7.5 mmol, 1.5 equiv) were added in sequence at −40 °C. The mixture was stirred at −40 °C for 36 h and detected by TLC. After completion, flash column chromatography was carried out to provide the desired product (**ent-3an)**. The product was used immediately for HPLC and NMR analysis.

9. Synthetic transformations of the product 3aa and ent-3an



A suspension of $[Ir(COD)(PCy_3)(Py)]PF_6$ (16 mg) and **3aa** (65 mg) in THF (3 mL) was stirred at 0 °C under 40 atm hydrogen atmosphere. After being stirred for 16 h, the mixture was filtrated through a pad of Celite and the filtration was concentrated in

vacuo, and there sidue was purified by column chromatography on silica gel to afford the desired product **5aa**.



5aa

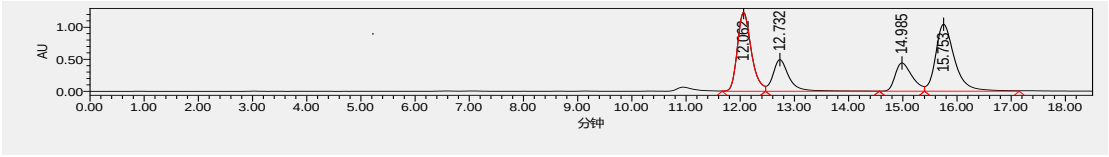
Methyl-(3aR,3bR,7aR,7bS)-1-((Z)-benzylidene)-4,7-dioxo-1,2,3,3a,4,7,7a,7b-octahydro-3bH-cyclopenta[3,4]cyclobuta[1,2]benzene-3b-carboxylate；

White oil; 88% yield, 99:1 e.r.,99:1d.r.; $[a]_D^{25}$ = −346.6 (c 1.08, CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IA, n-hexane/i-PrOH = 93/7, 1.0 mL/min, λ = 254 nm, t_1 = 12.07 min, t_2 = 12.75 min, t_3 = 15.04 min, t_4 = 15.69 min];

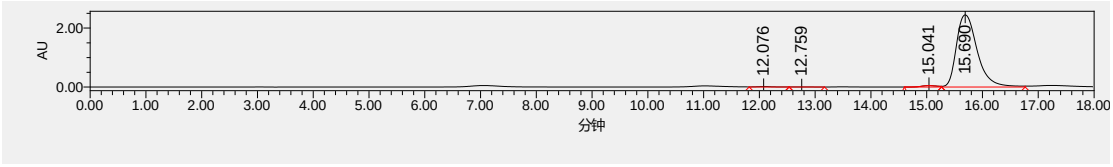
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, *J* = 7.6 Hz, 2H), 7.34 (t, *J* = 7.6 Hz, 2H), 7.22 (t, *J* = 7.2 Hz, 1H), 6.54 – 6.43 (m, 2H), 6.14 (d, *J* = 2.8 Hz, 1H), 4.06 (s, 1H), 3.84 – 3.80 (m, 2H), 3.77 (s, 3H), 3.14 – 2.96 (m, 2H), 2.93 – 2.77 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 206.2, 203.0, 167.7, 147.3, 144.5, 140.5, 136.5, 133.2, 128.4, 128.2, 127.1, 124.5, 100.0, 63.9, 54.8, 54.7, 53.1, 52.9, 39.9, 38.0.

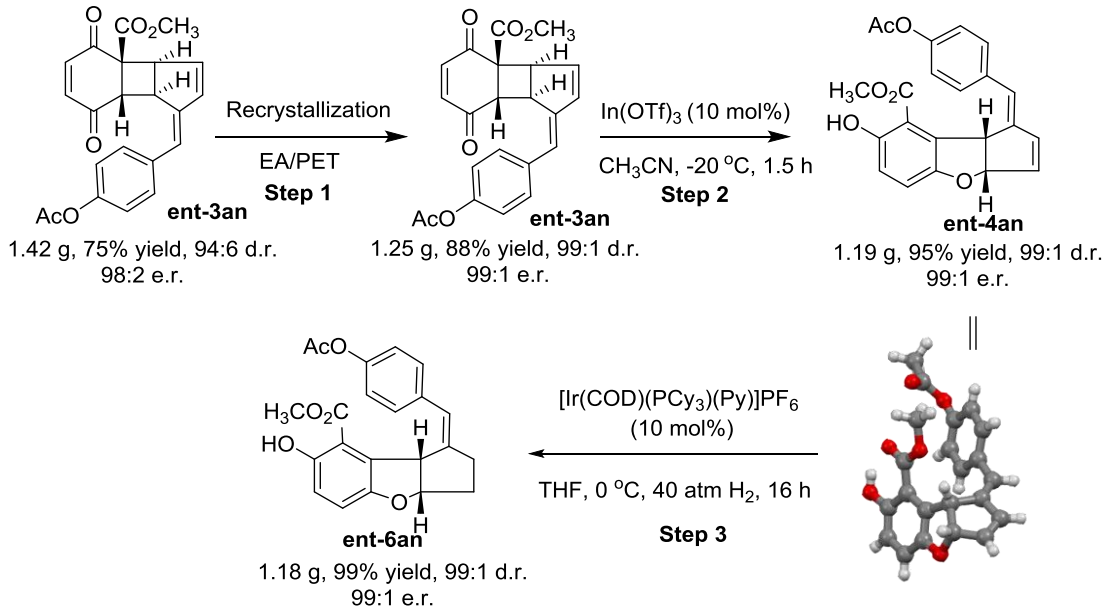
HRMS (ESI) calcd for $[M+H]^+$,$C_{20}H_{19}O_4$, m/z: 371.1283, observed: 371.1281;



	Retention Time	Area	% Area
1	12.062	21897322	32.61
2	12.732	10253021	15.27
3	14.985	9545626	14.22
4	15.753	25444063	37.90



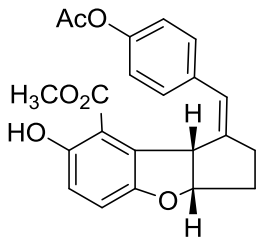
	Retention Time	Area	% Area
1	12.076	296885	0.47
2	12.759	66916	0.11
3	15.041	562611	0.88
4	15.690	62770797	98.55



Step 1: Recrystallization by using petroleum ether/EtOAc gave the product **ent-3an** in 88% yield with 99:1 d.r., 99:1 e.r..

Step 2:In a test tube with a magnetic stirring bar, **ent-3an** (3.3 mmol) in CH_3CN (60 mL) was cooled to −20 °C. $In(OTf)_3$ (0.33 mmol) in CH_3CN (13.0 mL) was stirred at 30 °C for 30 min. Then $In(OTf)_3$ (0. 33mmol) in CH_3CN (13.0 mL) was added in one-portion at −20 °C. The mixture was stirred at −20 °C for 1.5 h and detected by TLC. After completion, flash column chromatography was carried out to provide the desired product **ent-4an**.

Step 3:A suspension of $[Ir(COD)(PCy_3)(Py)]PF_6$ (250 mg) and **ent-4an** (1.19 g) in THF (30 mL) was stirred at 0 °C under 40 atm hydrogen atmosphere. After being stirred for 16 h, the mixture was filtrated through a pad of Celite and the filtration was concentrated in vacuo, and there sidue was purified by column chromatography on silica gel to afford the desired product **ent-6an**.



ent-6an

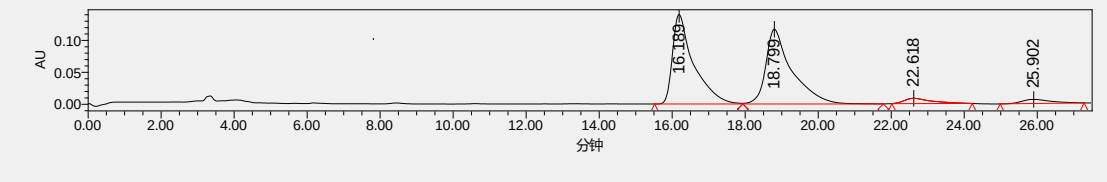
Methyl-(3aS,8bS)-1-((Z)-4-acetoxybenzylidene)-7-hydroxy-2,3,3a,8b-tetrahydro-1H-cyclopenta[b]benzofuran-8-carboxylate;

White oil; 99% yield, 99:1 e.r., 99:1d.r.; $[a]_D^{25}$ = 128.9 (c 0.95, CH_2Cl_2); Determined by HPLC analysis [Daicel chiralpak IC, n-hexane/i-PrOH = 90/10, 1.0 mL/min, λ = 254 nm, t_1 = 15.93 min, t_2 = 18.85 min,t_3 = 22.38 min,t_4 = 25.85 min];

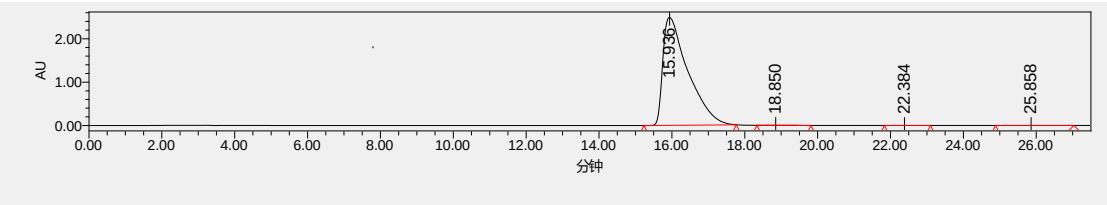
^1H NMR (400 MHz, $CDCl_3$) δ 10.23 (s, 1H), 7.21 (d, *J* = 8.4 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 1H), 6.78 (d, *J* = 8.8 Hz, 1H), 6.40 (s, 1H), 5.31 (t, *J* = 6.0 Hz, 1H), 5.20 (d, *J* = 7.2 Hz, 1H), 3.10 (s, 3H), 2.47 – 2.39 (m, 1H), 2.30 (s, 3H), 2.29 – 2.26 (m, 1H), 2.12 -2.09 (m, 1H), 1.99 – 1.92 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.2, 169.6, 155.6, 153.8, 149.3, 144.9, 141.1, 135.7, 129.5, 127.6, 123.1, 121.7, 117.6, 116.3, 110.5, 87.4, 52.1, 49.4, 34.3, 33.5, 21.1.

HRMS (ESI) calcd for $[M+H]^+$,$C_{22}H_{20}O_6Na$, m/z: 381.1338, observed: 381.1694;



	Retention Time	Area	% Area
1	16.189	5608032	46.01
2	18.799	5796700	47.55
3	22.618	430843	3.53
4	25.902	354474	2.91



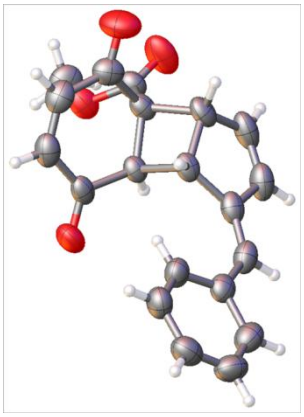
	Retention Time	Area	% Area
1	15.936	118756119	99.85
2	18.850	27509	0.02
3	22.384	14744	0.01
4	25.858	141946	0.12

10.X-ray crystalstructure of the product 3aa and ent-4an

X-ray crystals structure of the product 3aa:

Empirical formula	$C_{20}H_{16}O_4$
Formula weight	320.33
Temperature/K	294.39(10)
Crystal system	monoclinic
Space group	C2
a/Å	16.8740(4)
b/Å	8.2225(2)
c/Å	11.5745(3)
α/°	90
β/°	92.962(2)
γ/°	90
Volume/Å^3	1603.79(7)
Z	4
ρ_{calc}g/cm^3	1.327
μ/mm^{-1}	0.754
F(000)	672.0
Crystal size/mm^3	0.8 ×0.5 ×0.2

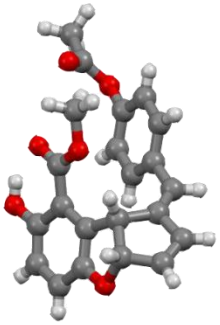
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	10.5 to 134.152
Index ranges	$-20 \leq h \leq 19$, $-9 \leq k \leq 9$, $-13 \leq l \leq 13$
Reflections collected	8300
Independent reflections	2782 [R_{int} = 0.0293, R_{sigma} = 0.0288]
Data/restraints/parameters	2782/1/218
Goodness-of-fit on F^2	1.074
Final R indexes [I>=2σ (I)]	R_1 = 0.0508, wR_2 = 0.1295
Final R indexes [all data]	R_1 = 0.0517, wR_2 = 0.1316
Largest diff. peak/hole / e $Å^{-3}$	0.42/-0.24
Flack parameter	-0.08(12)



X-ray crystal structure of product ent-4an:

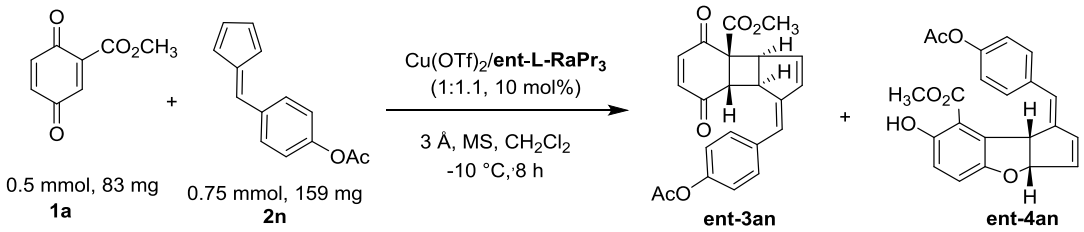
Empirical formula	$C_{22}H_{18}O_6$
Formula weight	378.36
Temperature/K	125
Crystal system	monoclinic
Space group	$P2_1$
a/Å	7.67071(16)
b/Å	13.2339(2)
c/Å	9.6030(2)
α/°	90
β/°	112.936(3)
γ/°	90
Volume/$Å^3$	897.77(4)
Z	2
$\rho_{calc}g/cm^3$	1.400
μ/mm^{-1}	0.850
F(000)	396.0
Crystal size/mm^3	0.8 ×0.5 ×0.3

Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	10.002 to 134.11
Index ranges	$-7 \leq h \leq 9, -15 \leq k \leq 15, -11 \leq l \leq 9$
Reflections collected	8864
Independent reflections	3210 [R_{int} = 0.0244, R_{sigma} = 0.0241]
Data/restraints/parameters	3210/1/256
Goodness-of-fit on F^2	1.063
Final R indexes [I>=2σ (I)]	R_1 = 0.0430, wR_2 = 0.1124
Final R indexes [all data]	R_1 = 0.0433, wR_2 = 0.1129
Largest diff. peak/hole / e $Å^{-3}$	0.32/-0.31
Flack parameter	0.03(7)

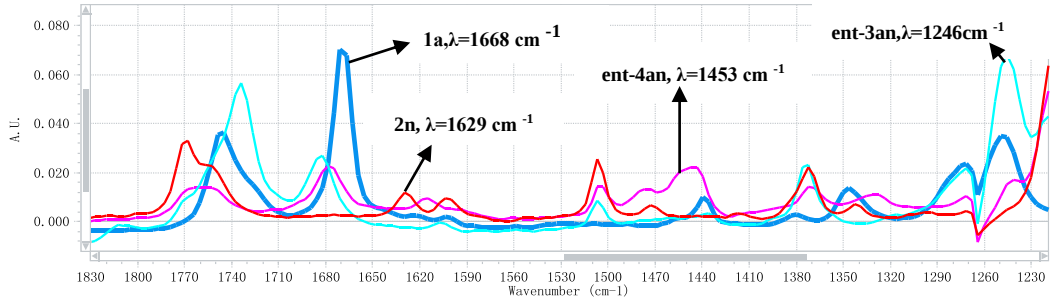


11. Operando IR experiments

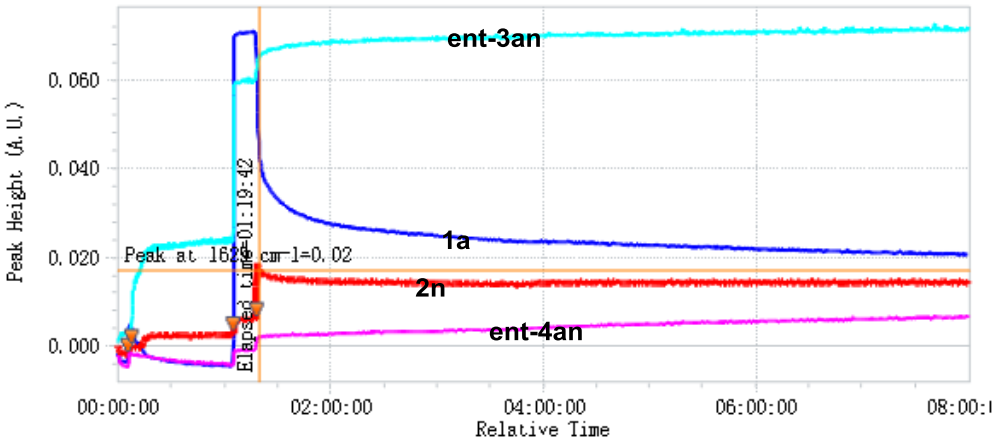
Static stateand Operando IR experiments:



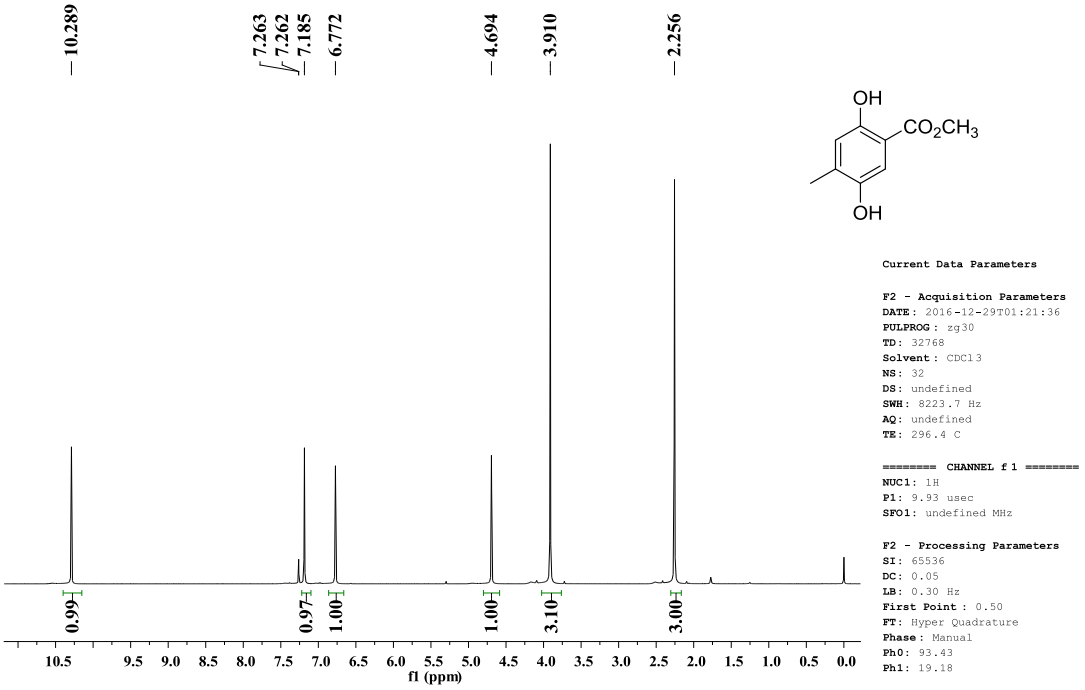
Step 1: We initiated our studies by detecting the static state spectrogram of the substrates **1a**, **2n**, and the products **ent-3an**, **ent-4an**.The static state spectrogram and the characteristic IR peaks of **1a**, **2n, ent-3an** and **ent-4an** were established.

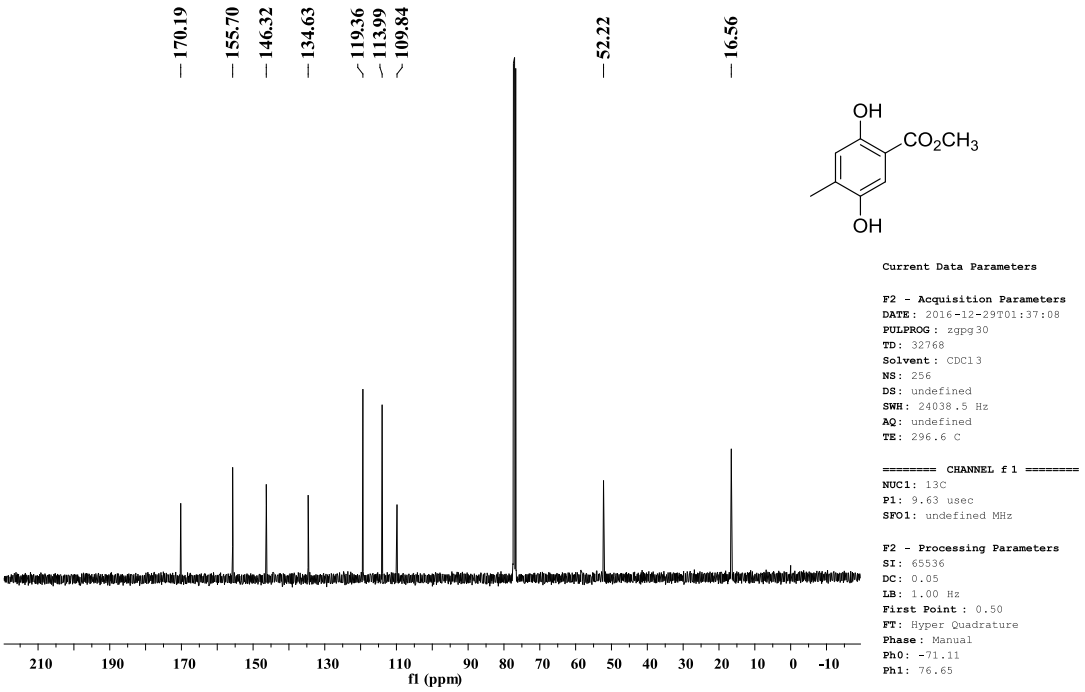


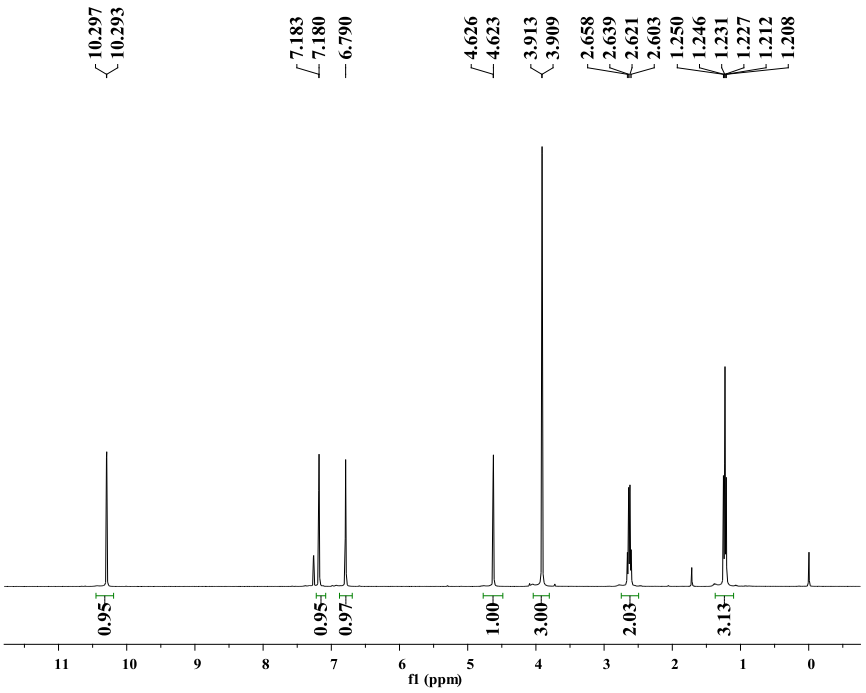
Step 2: We did Operando IR experimentsand detected the characteristic IR peaks (**1a**, **2n, ent-3ao** and **ent-4an**). From the spectrogram we could obtain the variation trend of different compound. As illustrated in the spectrogram, when the substrate **1a** was added the absorption at 1668 cm^{-1}appeared (blue line). After the substrate **2n** was added (redline), the reaction begun. As the peaks at 1668 and 1629cm^{-1} related to the substrate **1a** and **2n** depleted gradually, the peaks of the product **ent-3an** at 1246 cm^{-1}(greenline) and **ent-4an** at 1453cm^{-1}(purpleline) increased. It indicates that product **ent-3an** and **ent-4an** generate at same time instead of the product **ent-3an** transform to **ent-4an.**

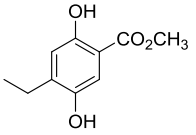


12. NMR spectra









Current Data Parameters

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TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.4 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
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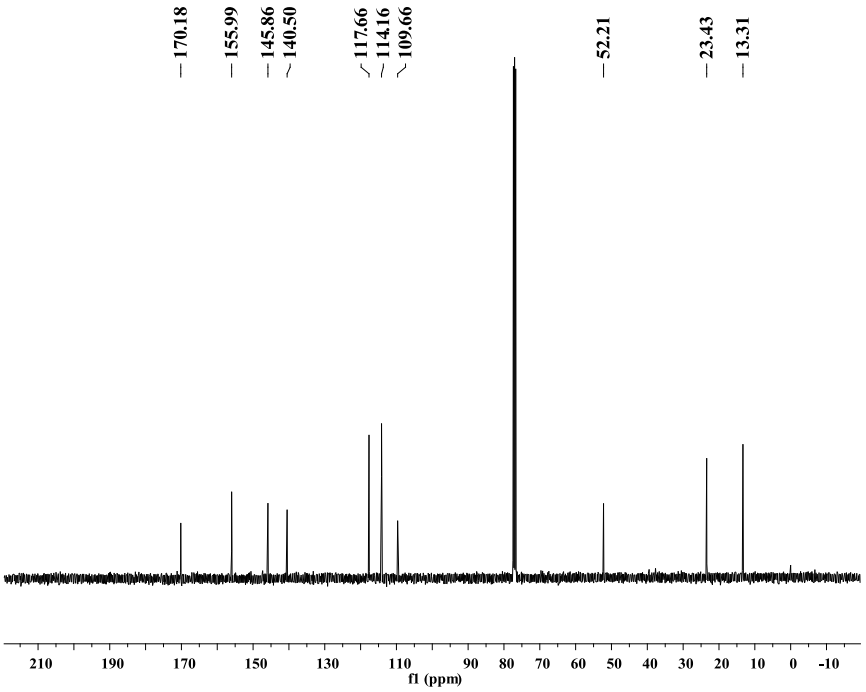
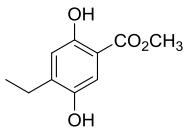
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 93.84
Ph1: 18.05





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Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.7 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
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LB: 1.00 Hz
First Point: 0.50
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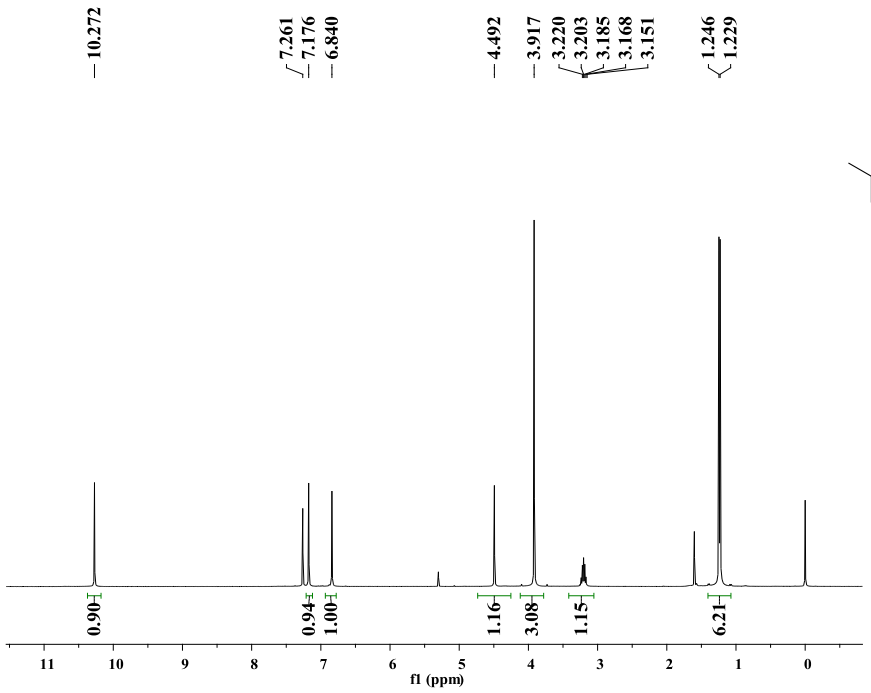
Ph1: 42.33



Current Data Parameters

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Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.3 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
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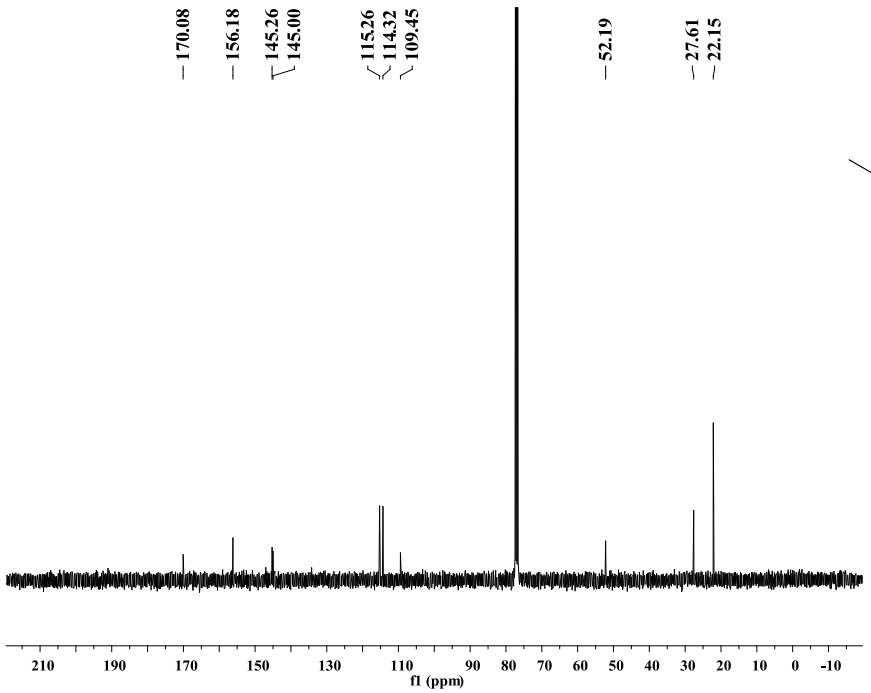
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 90.77
Ph1: 19.20



Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-29T02:20:12
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.5 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
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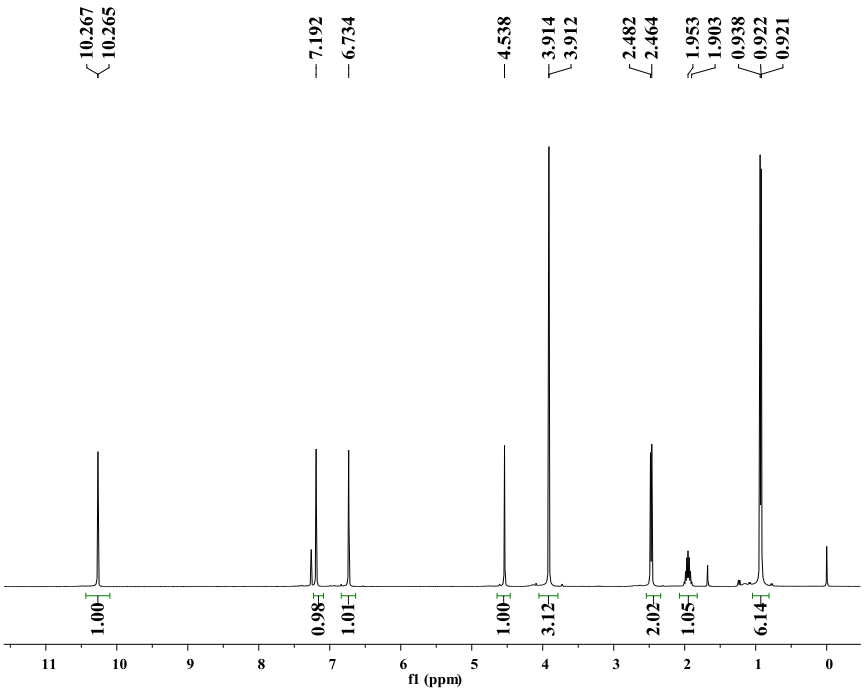
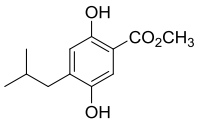
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -27.93
Ph1: -6.86





Current Data Parameters

F2 - Acquisition Parameters
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NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.3 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
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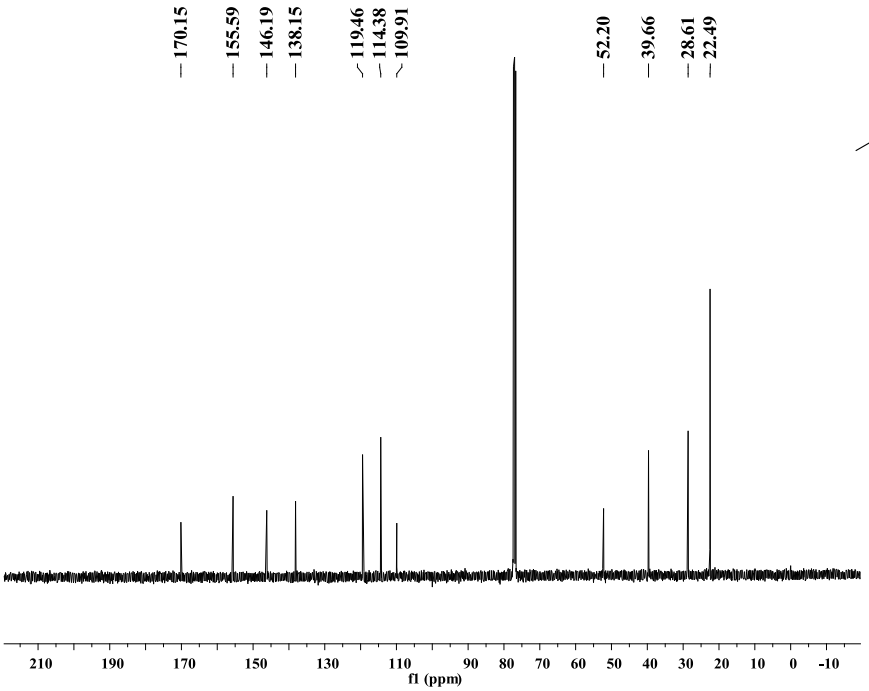
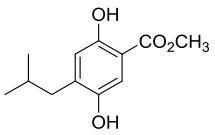
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -265.74
Ph1: 16.44





Current Data Parameters

F2 - Acquisition Parameters
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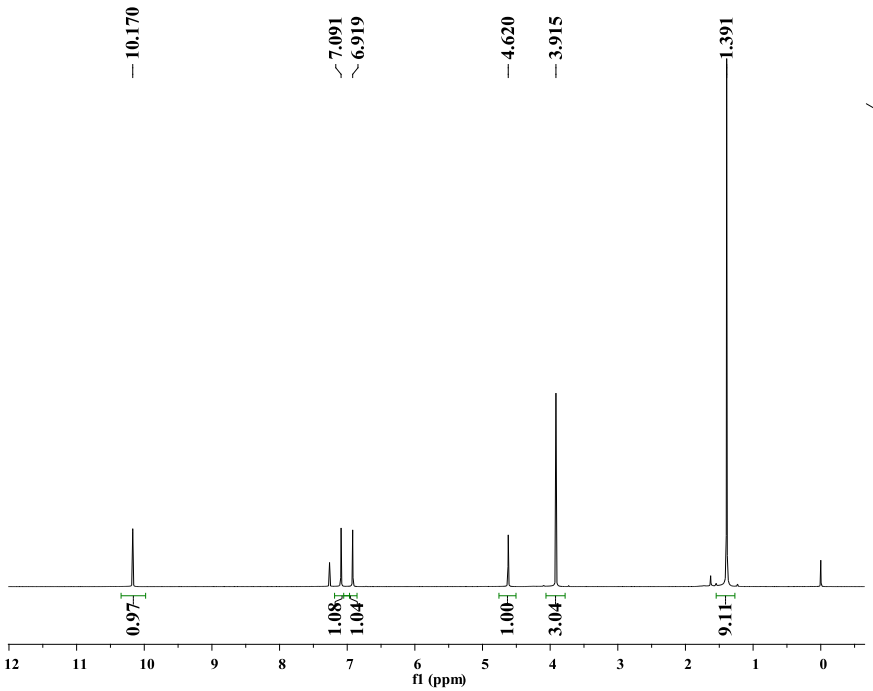
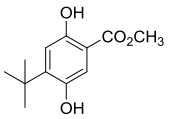
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.6 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -85.69
Ph1: 93.58



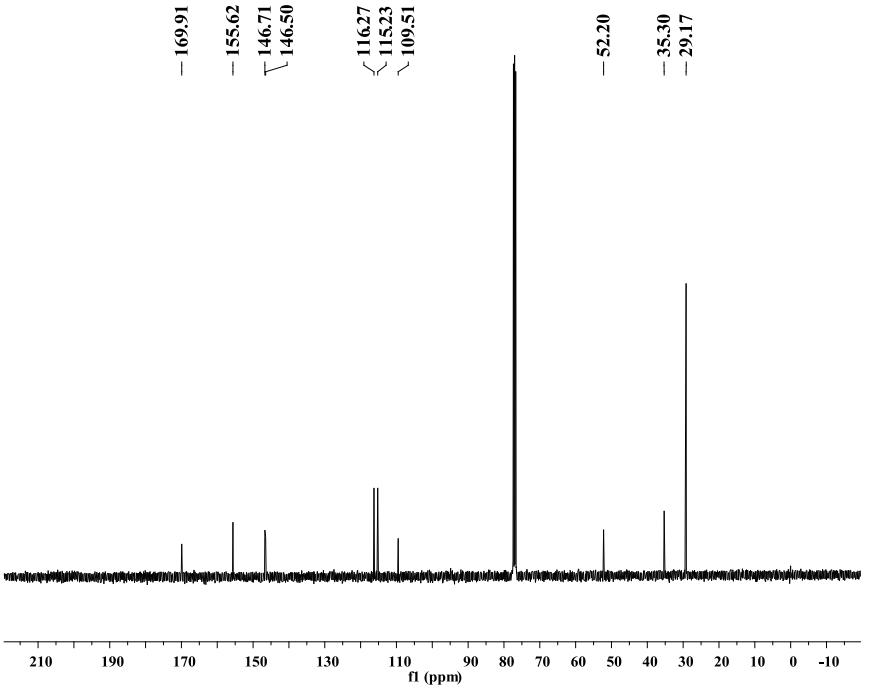


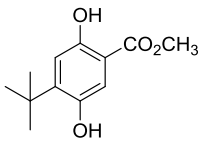
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-29T02:47:47
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.4 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 93.70
Ph1: 18.93



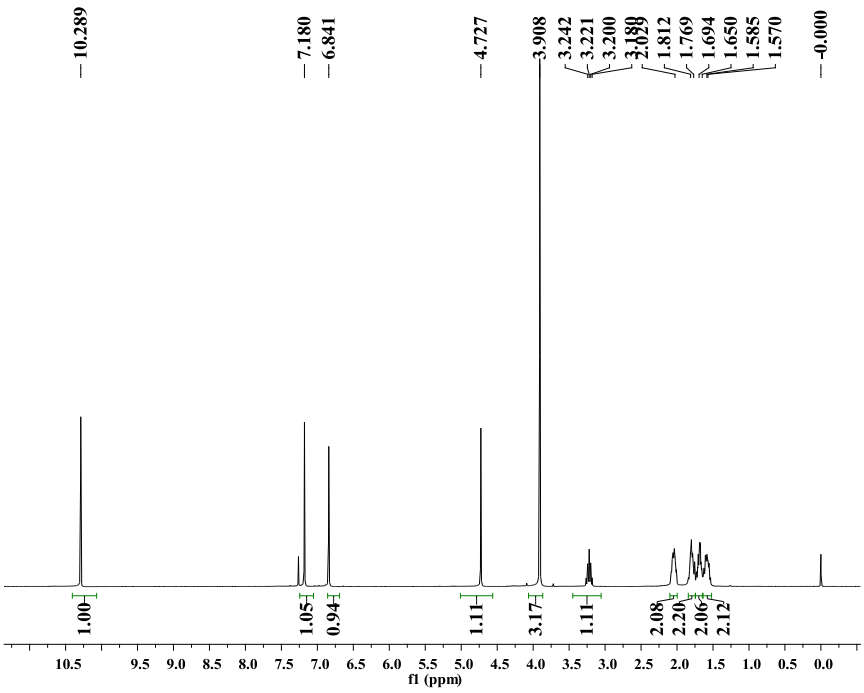


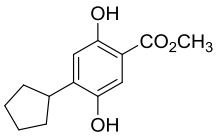
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-29T03:03:20
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.6 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -69.77
Ph1: 75.94



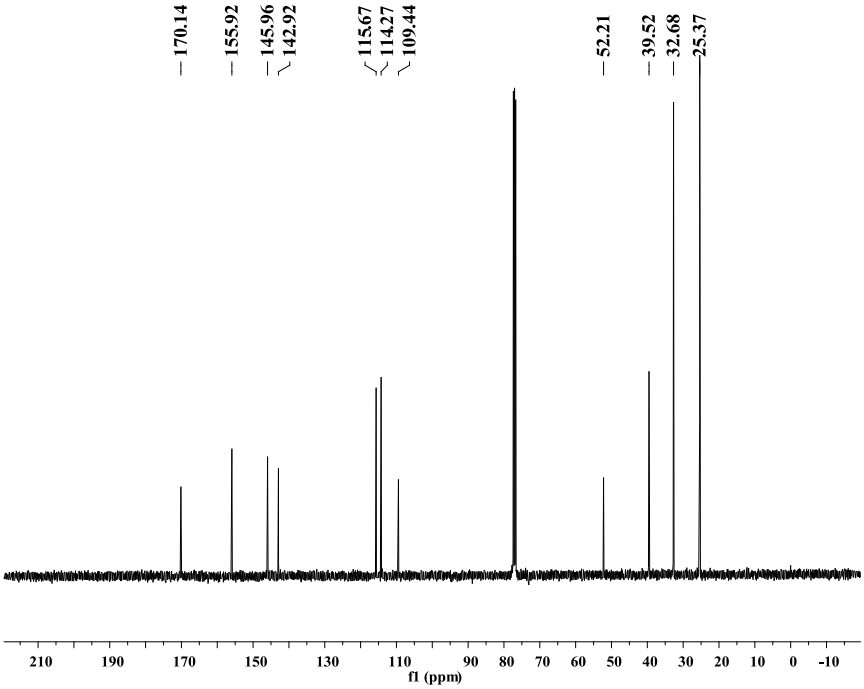


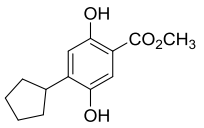
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-29T12:25:31
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.1 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 96.69
Ph1: 13.04



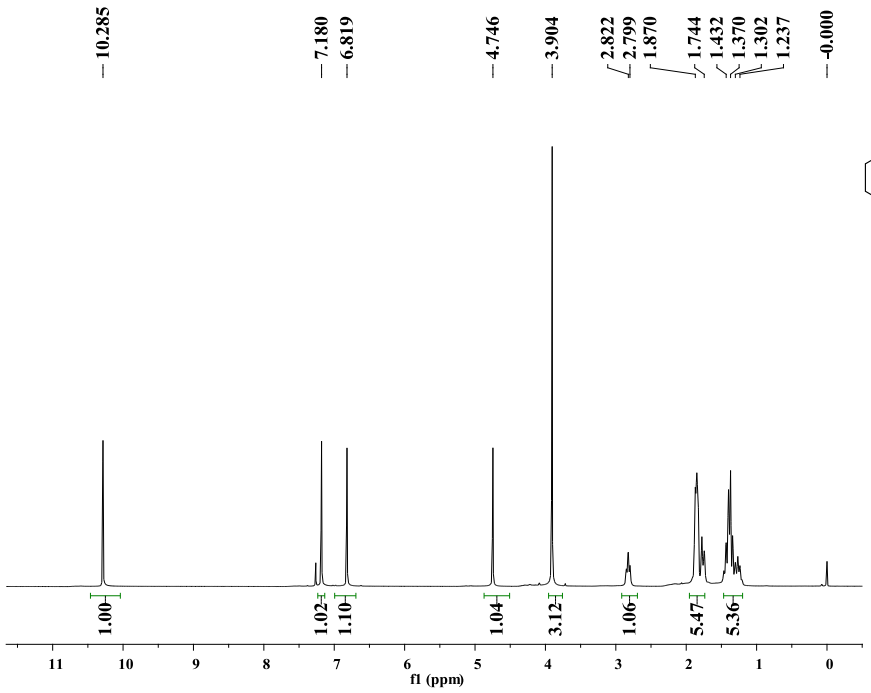


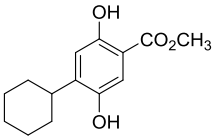
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-29T12:41:04
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.4 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -70.17
Ph1: 73.85



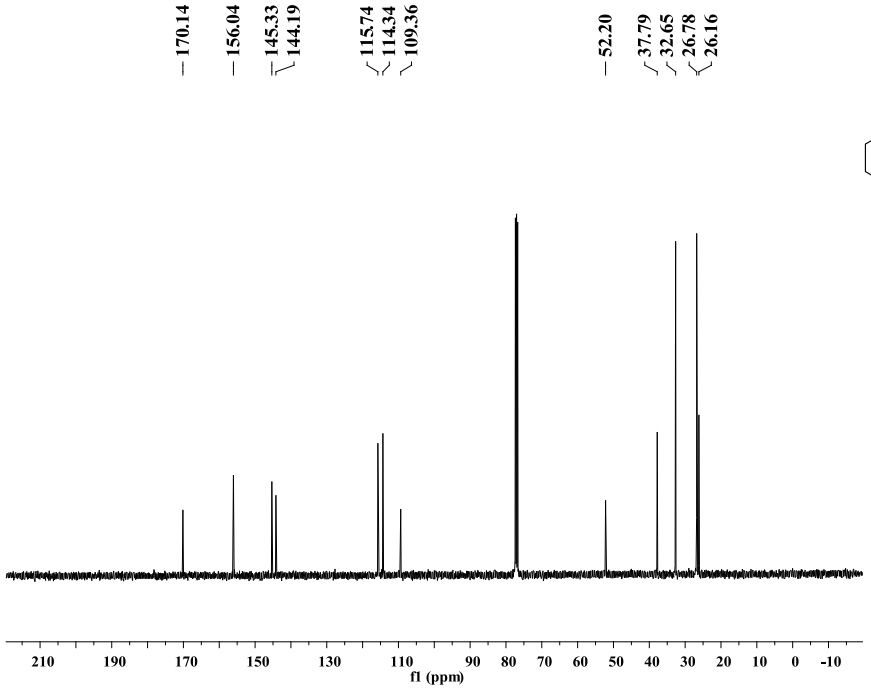


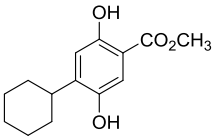
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-29T03:09:30
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.2 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 81.04
Ph1: 34.52



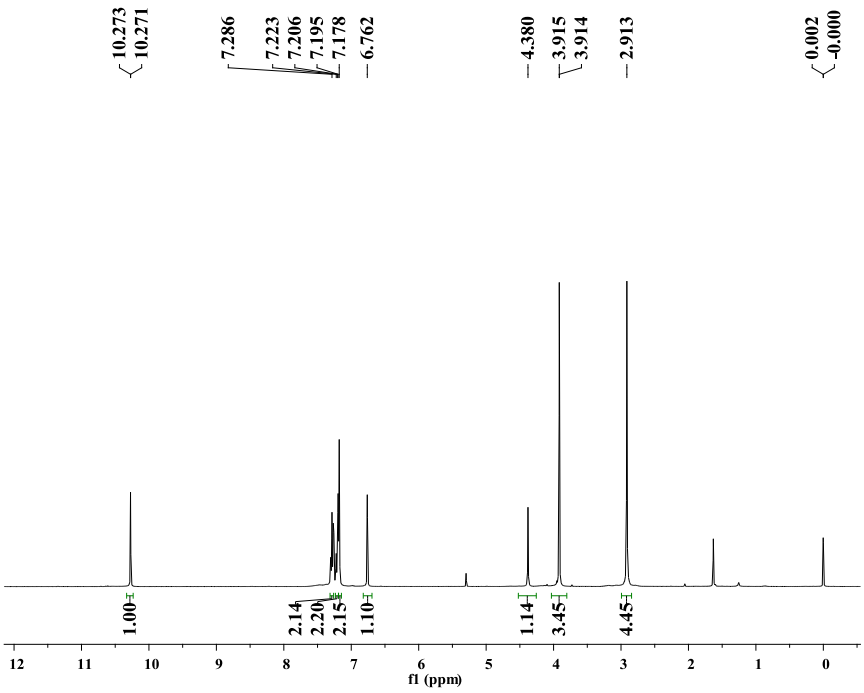


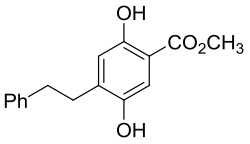
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-29T03:25:03
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.5 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -86.21
Ph1: 90.55



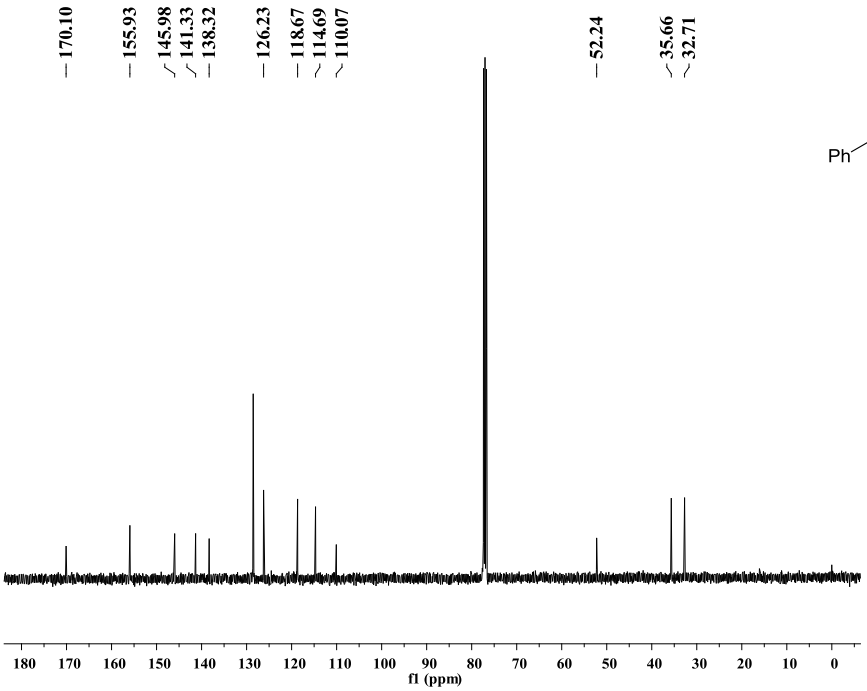


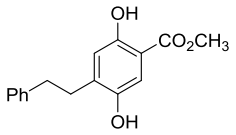
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-29T03:31:13
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.3 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 91.99
Ph1: 18.86



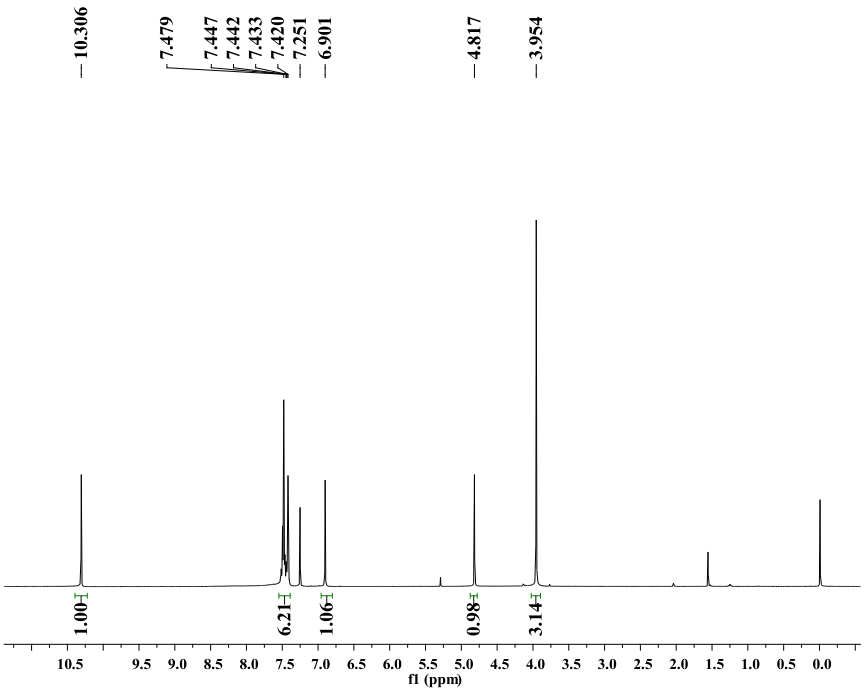


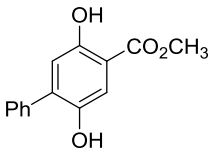
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-29T03:46:45
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.5 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -61.74
Ph1: 48.21



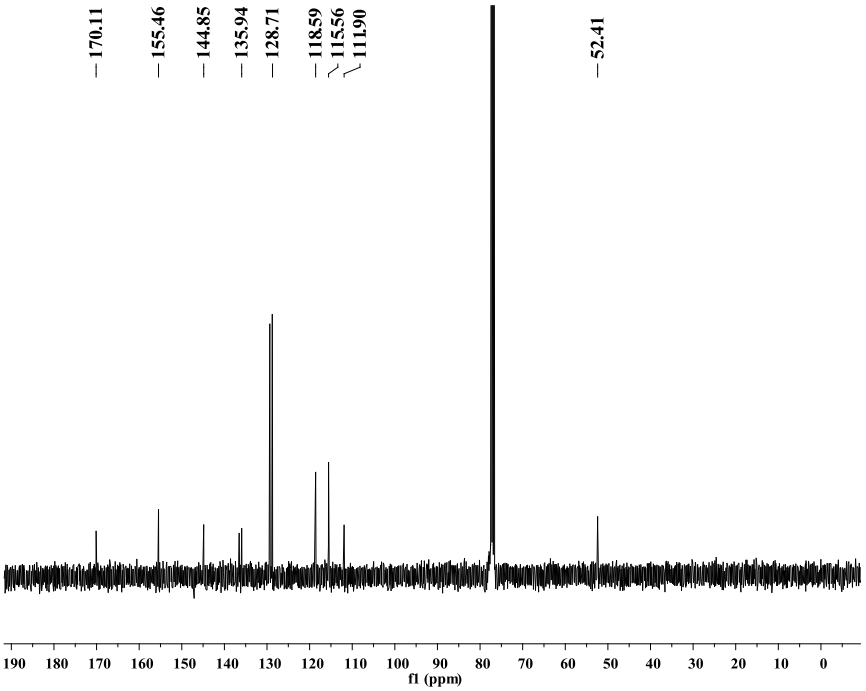


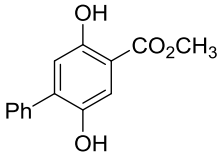
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-28T23:11:35
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.2 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 103.30
Ph1: 5.79



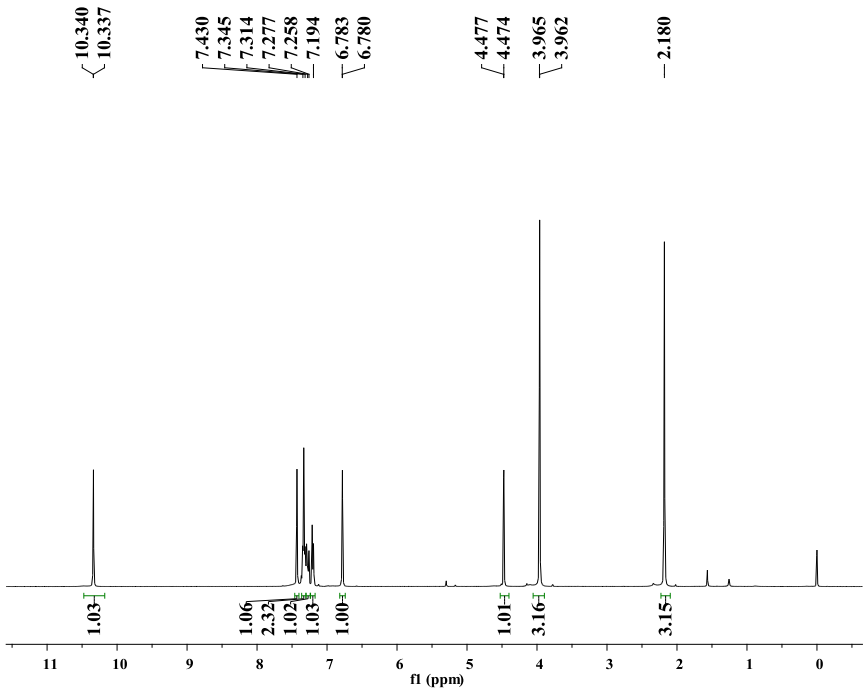


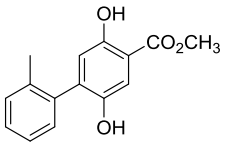
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-28T23:27:13
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.5 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -47.38
Ph1: 24.38



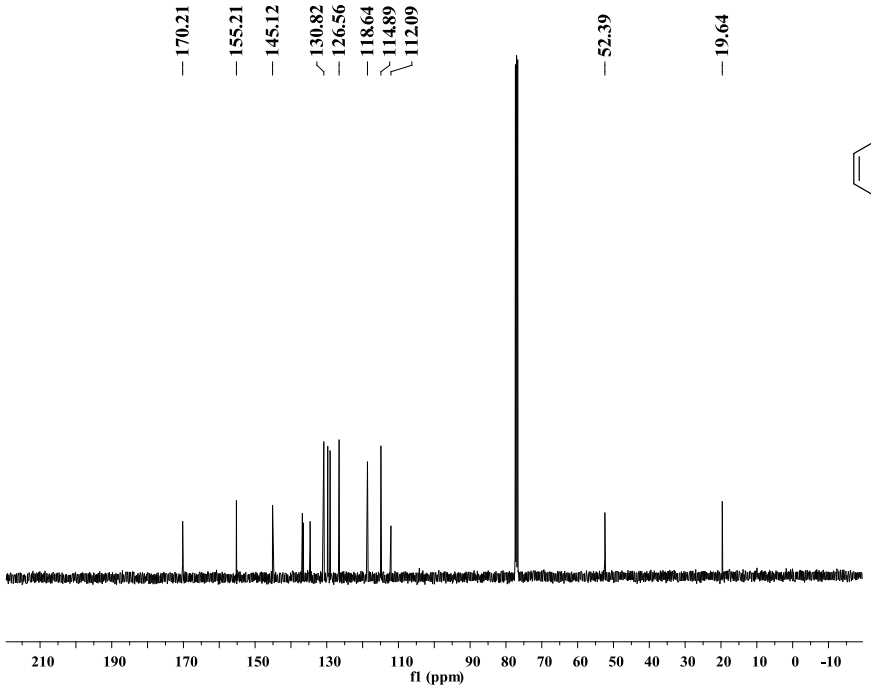


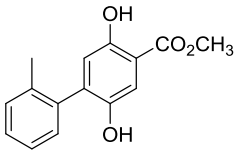
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-28T23:33:12
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.2 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 91.08
Ph1: 21.69



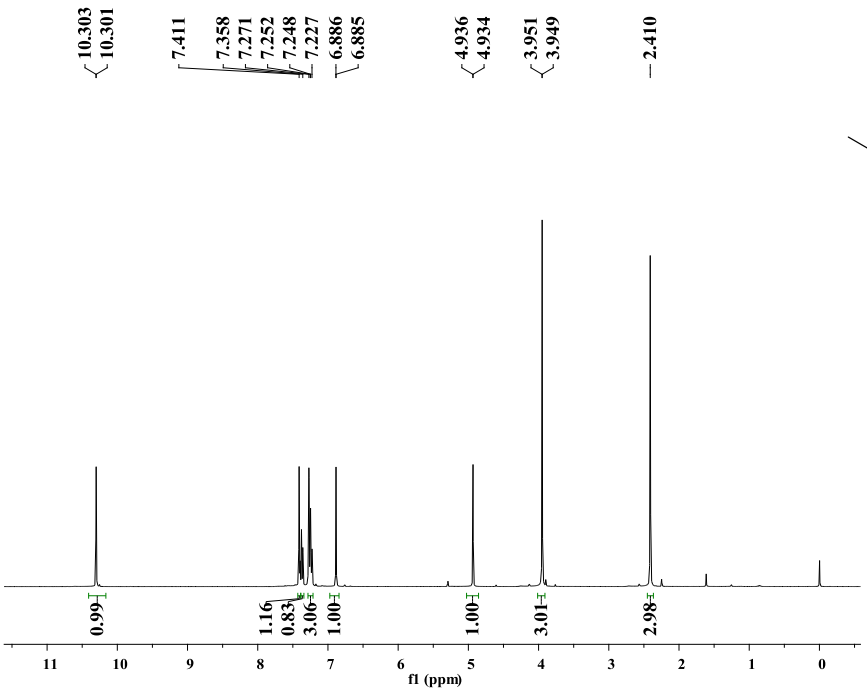


Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-28T23:48:44
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.6 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -69.70
Ph1: 67.78

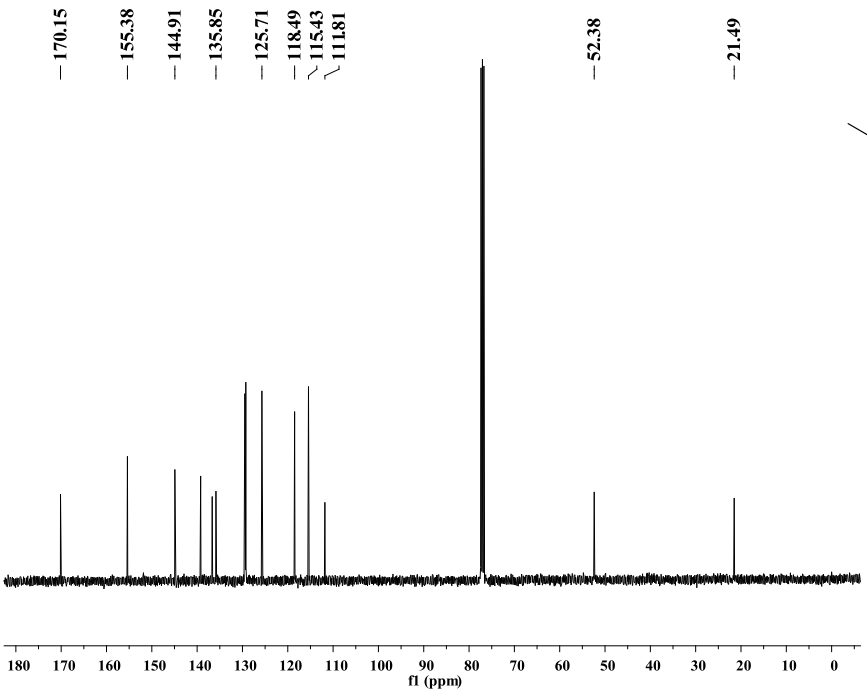


Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-28T23:54:28
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.4 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 92.06
Ph1: 20.54

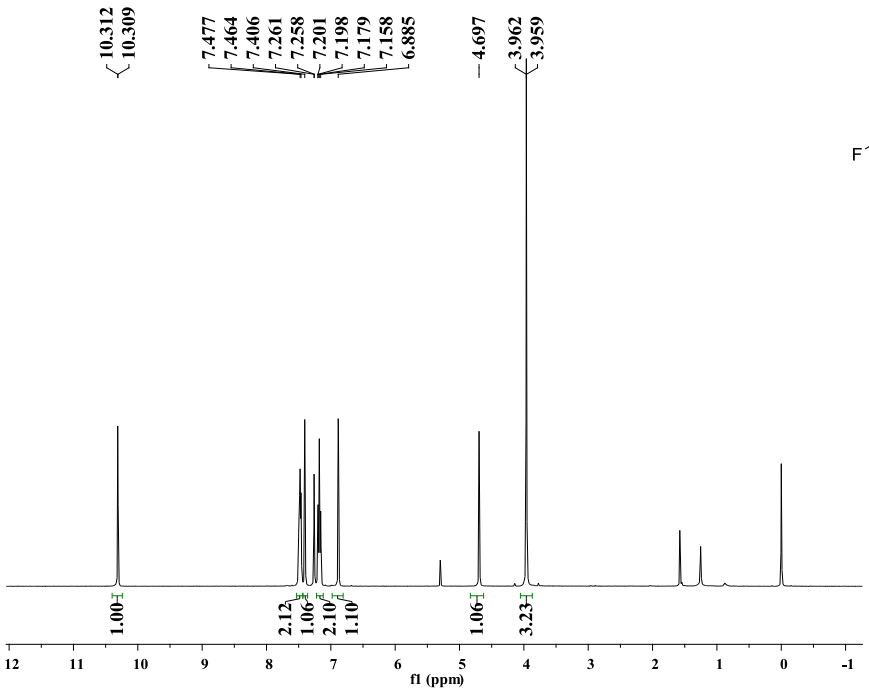


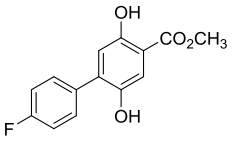
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-29T00:10:00
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.7 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -68.79
Ph1: 71.14



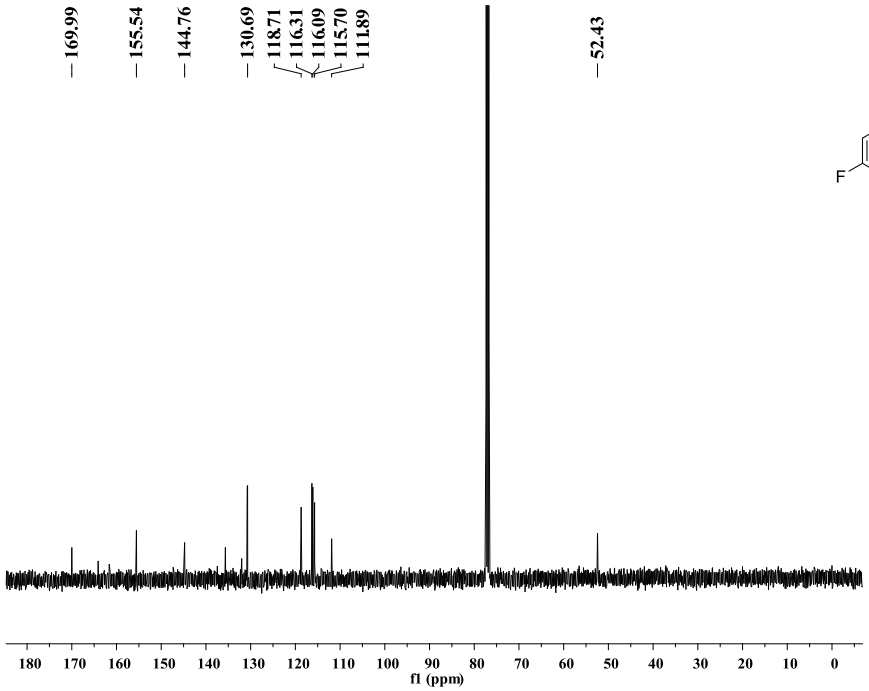


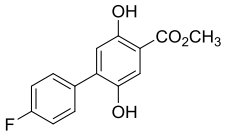
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-29T00:38:38
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.4 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 94.12
Ph1: 16.86



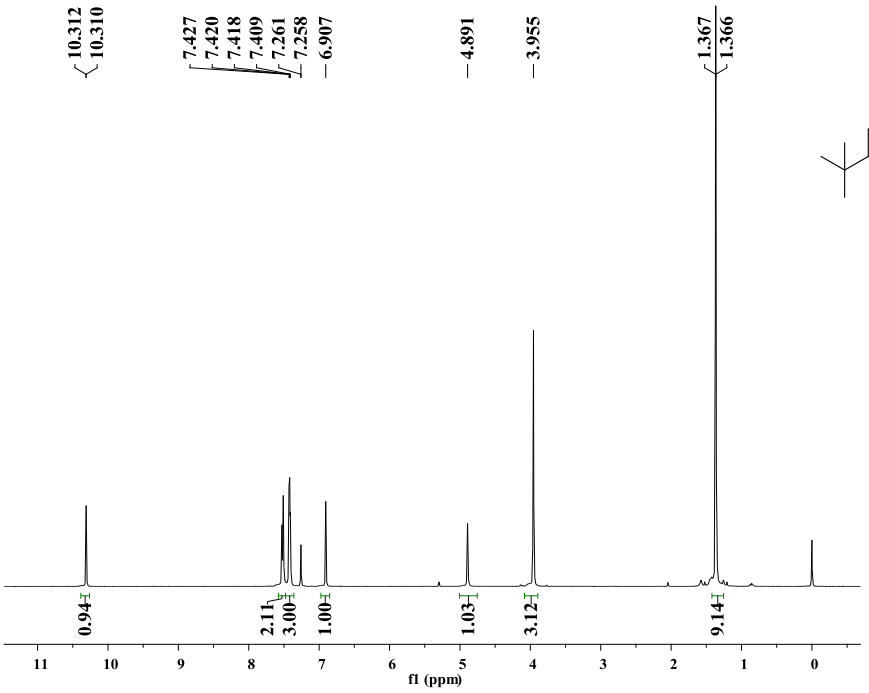


Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-29T00:54:10
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.8 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -52.88
Ph1: 40.49

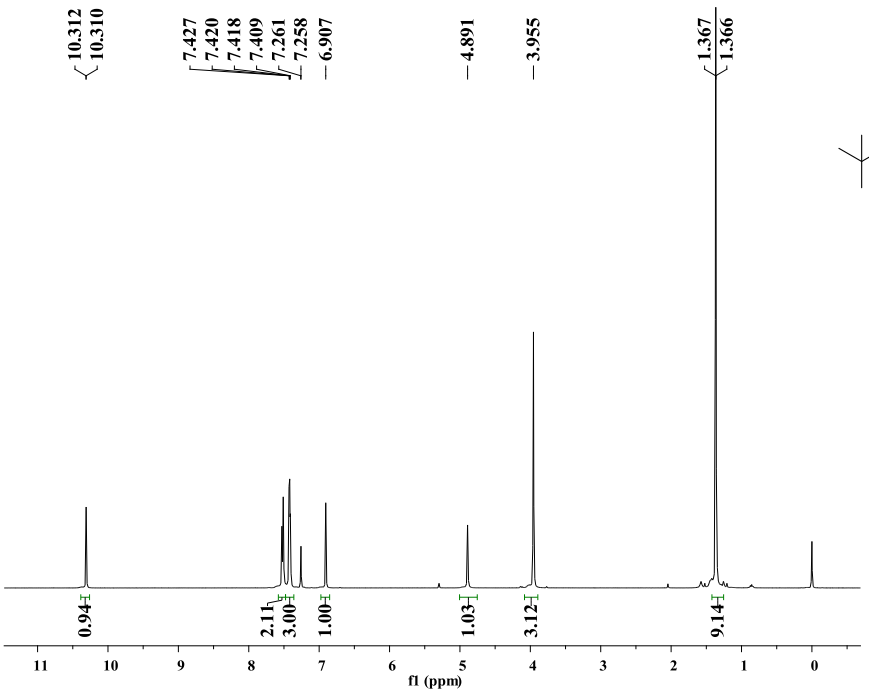


Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-29T00:16:07
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.2 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 92.11
Ph1: 19.88

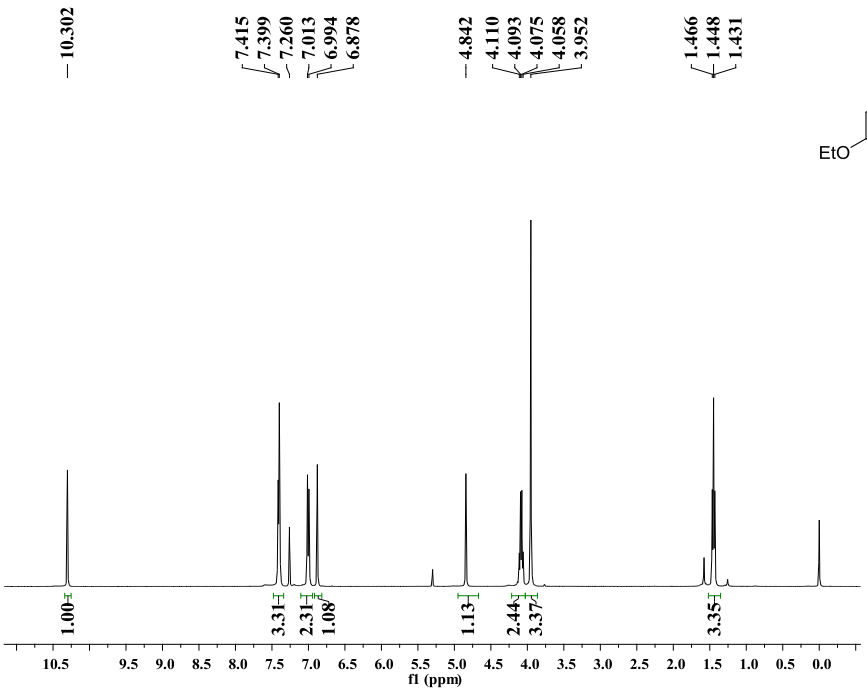


Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-29T00:16:07
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.2 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 92.11
Ph1: 19.88

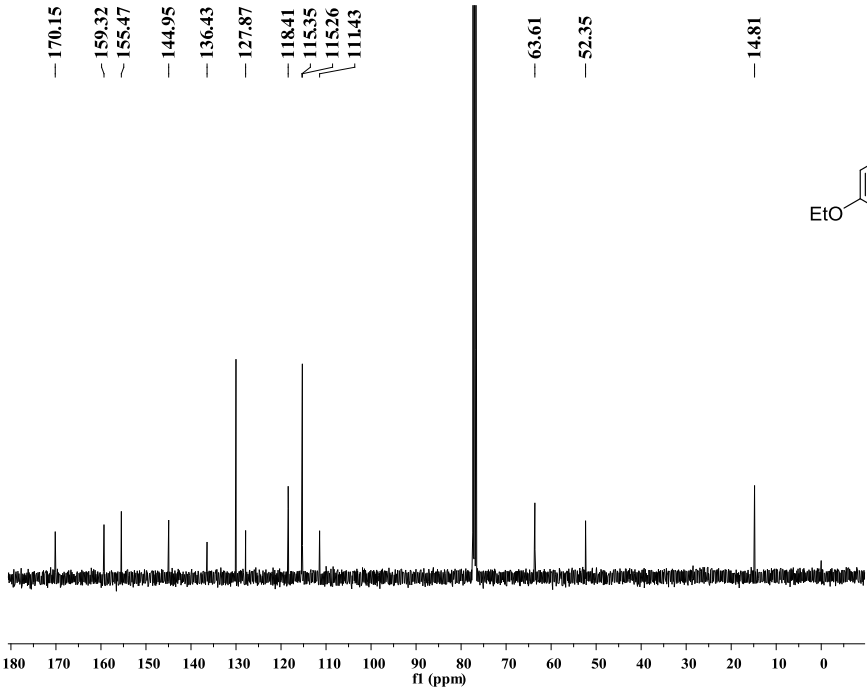


Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-29T00:59:48
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.5 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 91.53
Ph1: 19.44

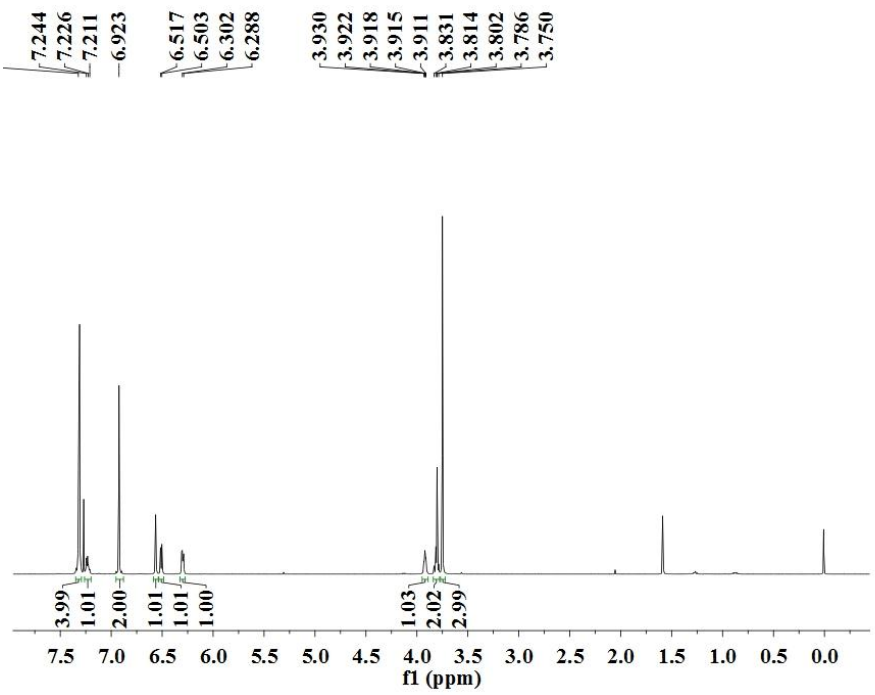


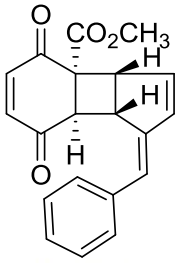
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-29T01:15:20
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.7 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -53.31
Ph1: 36.99



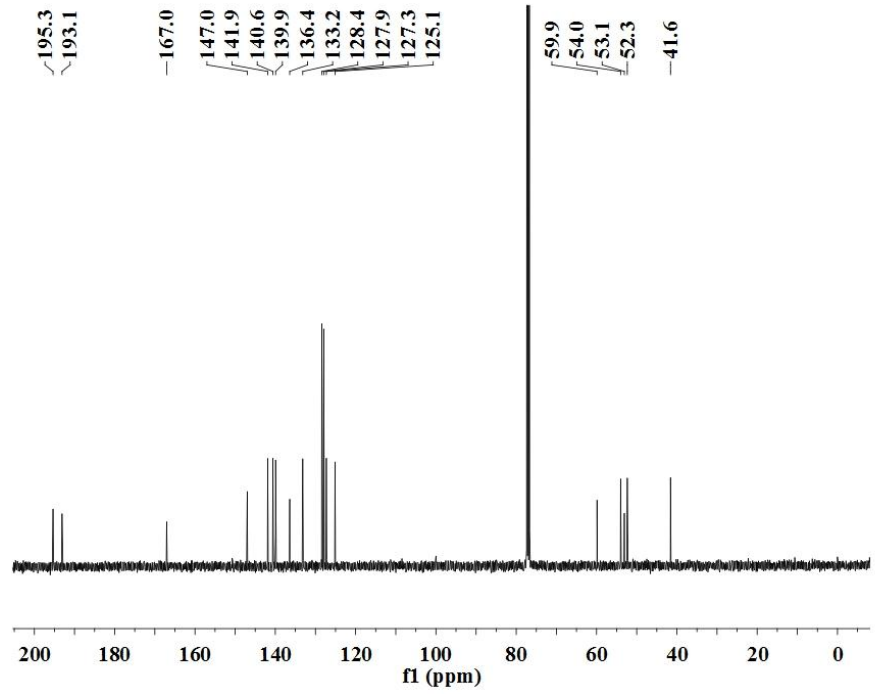


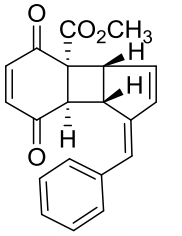
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2015-11-17T23:24:21
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 94.89
Ph1: 17.26



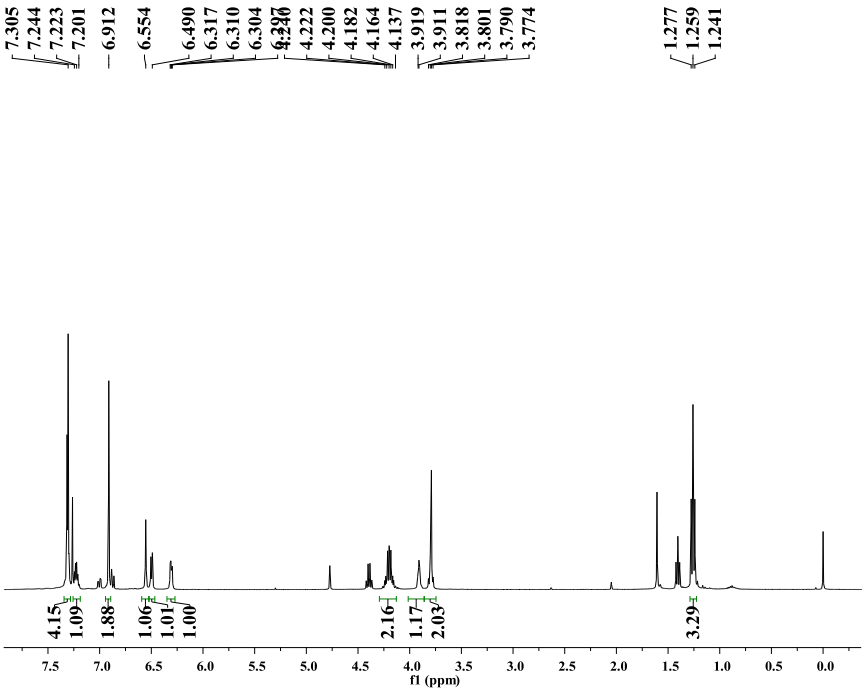


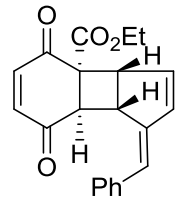
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2015-11-17T23:54:33
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 512
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -70.74
Ph1: 62.65



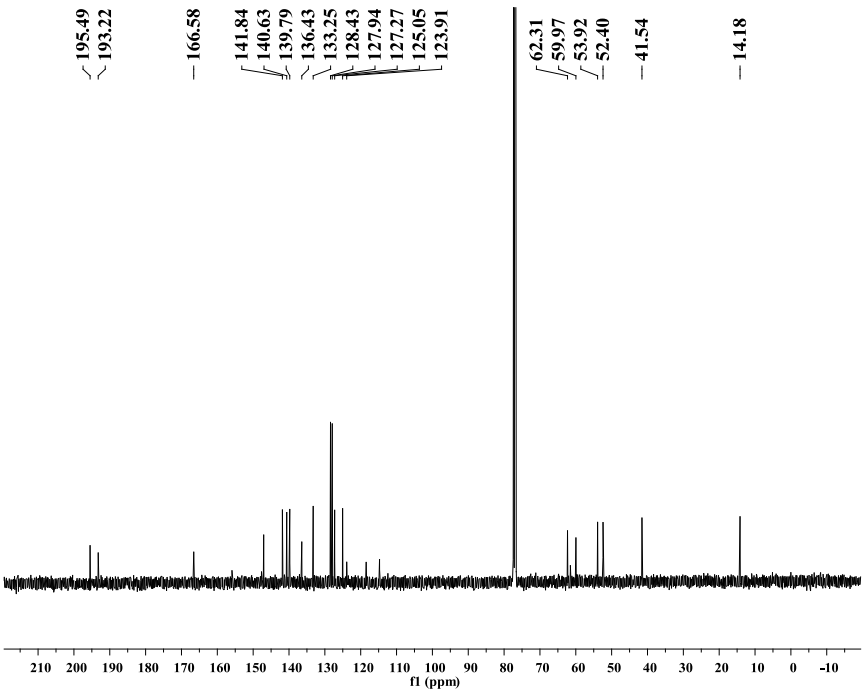


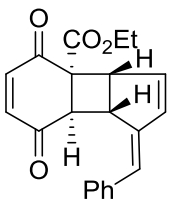
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-11-04T12:44:29
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295.2 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 98.17
Ph1: 11.63



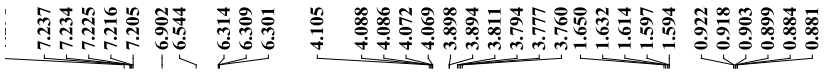


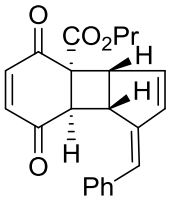
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-11-04T13:00:04
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 295.6 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -71.06
Ph1: 73.44



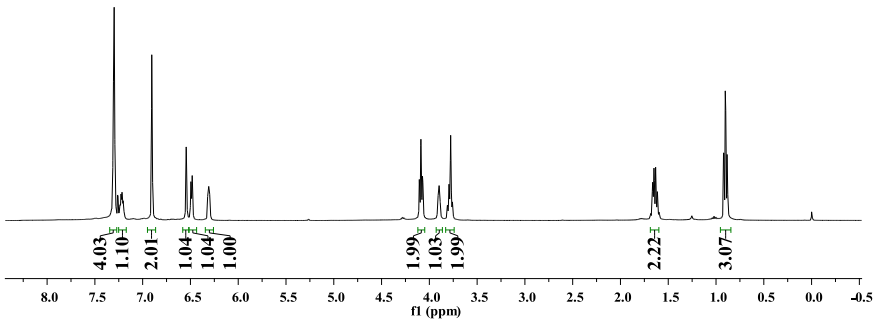


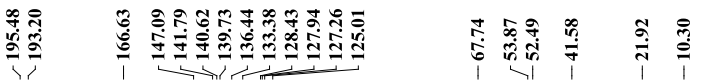
Current Data Parameters

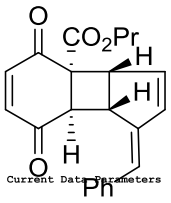
F2 - Acquisition Parameters
DATE: 2016-11-03T16:13:04
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295.4 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 100.94
Ph1: 13.48





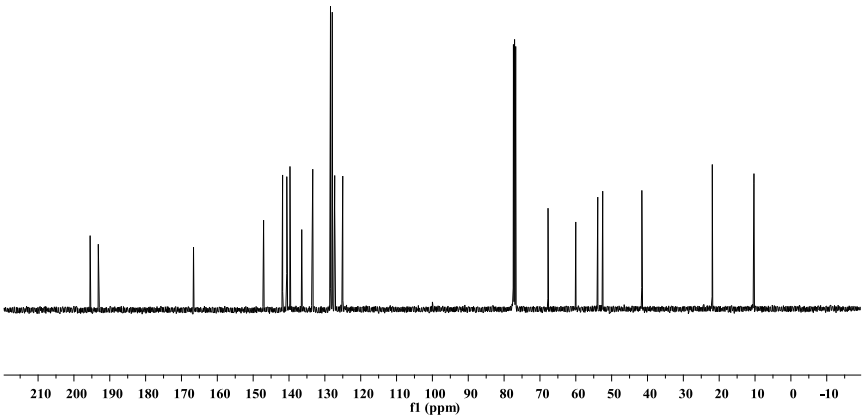


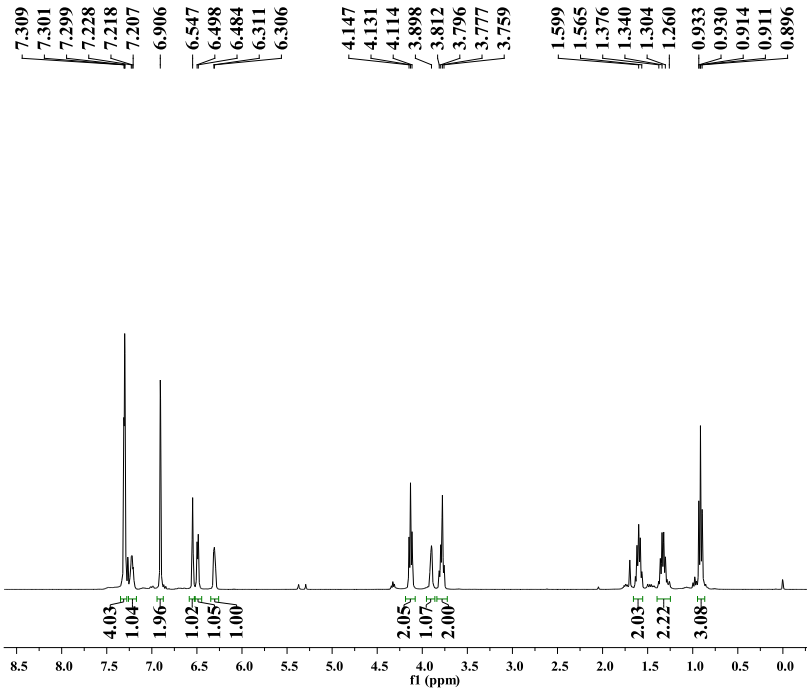
Current Data Parameters

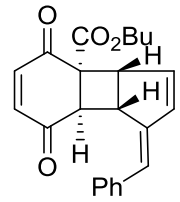
F2 - Acquisition Parameters
DATE: 2016-11-03T17:04:32
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 295.6 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -80.74
Ph1: 86.89





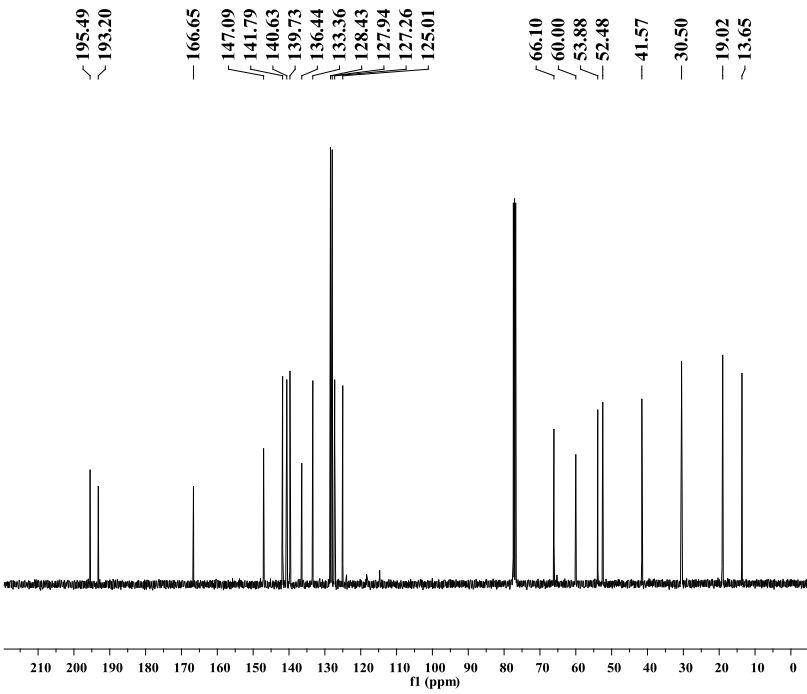


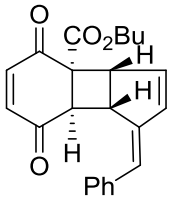
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-11-03T16:18:57
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295.3 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -261.19
Ph1: 20.11



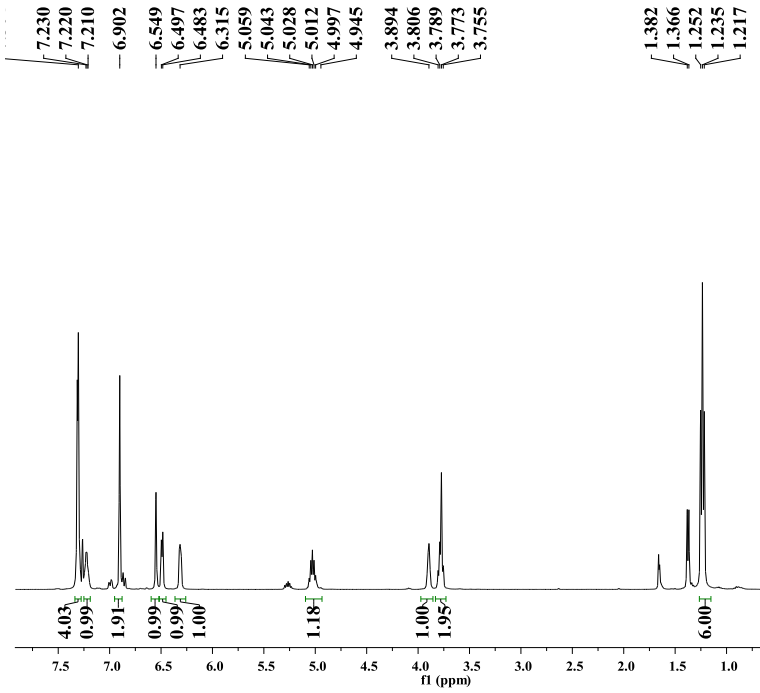


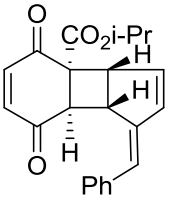
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-11-03T17:22:42
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 295.7 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -67.85
Ph1: 58.72



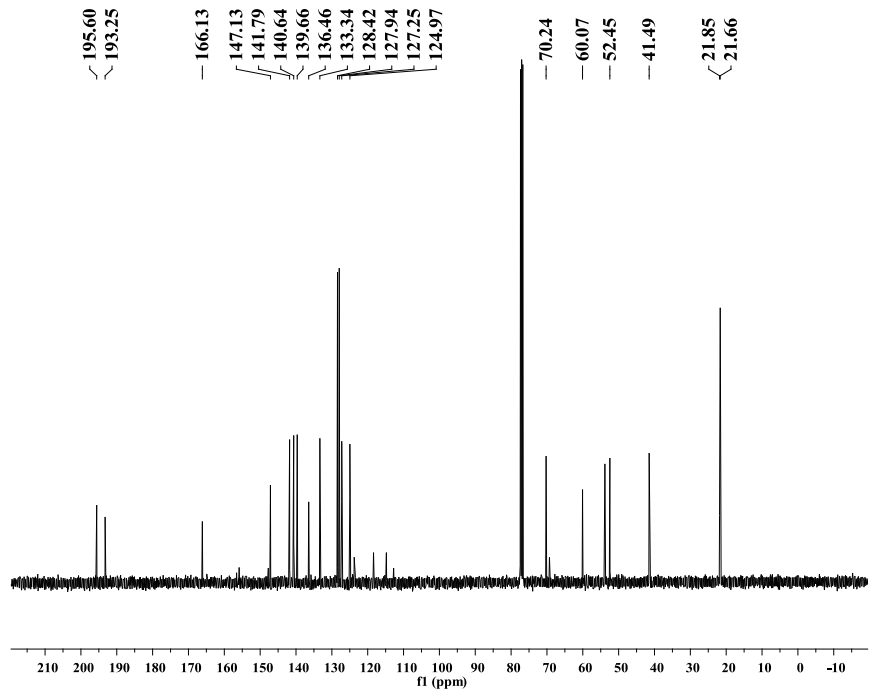


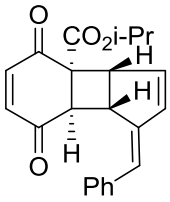
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-11-03T16:25:45
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295.3 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -261.22
Ph1: 19.86



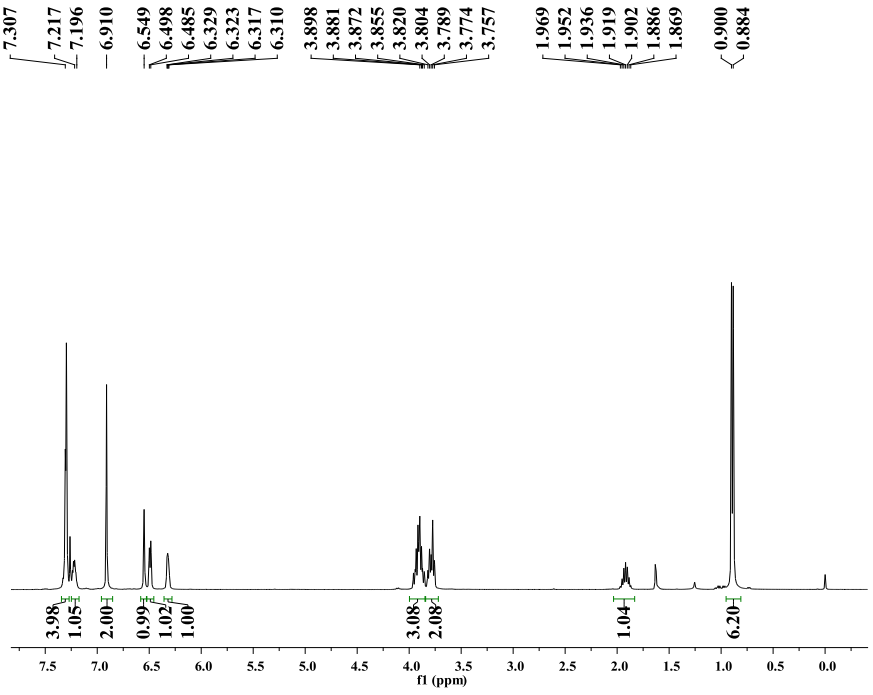


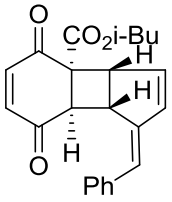
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-11-03T17:41:39
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 295.8 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -51.73
Ph1: 37.48



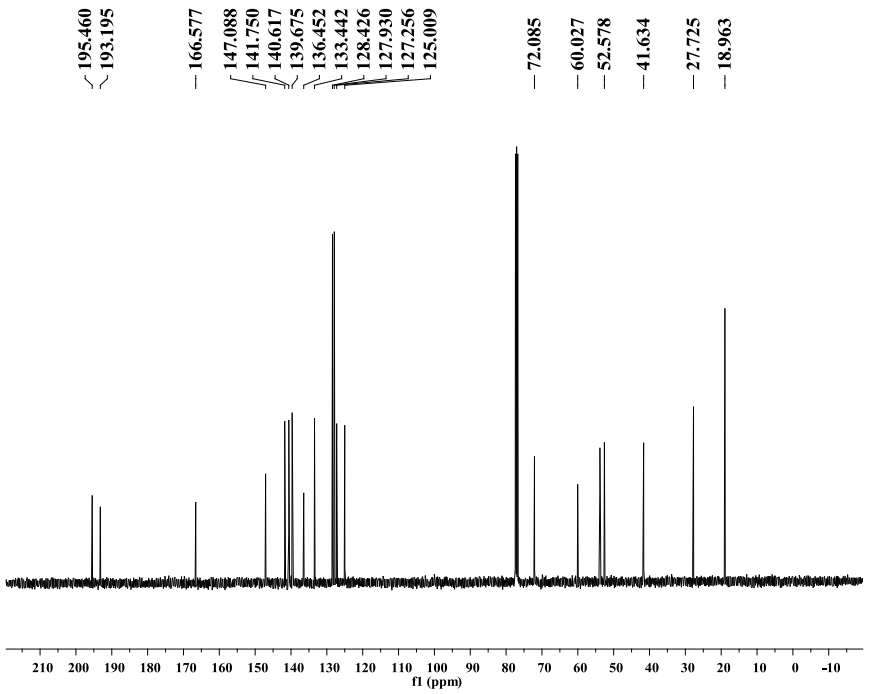


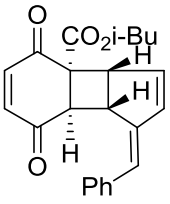
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-11-03T16:32:29
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295.4 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -262.75
Ph1: 19.77



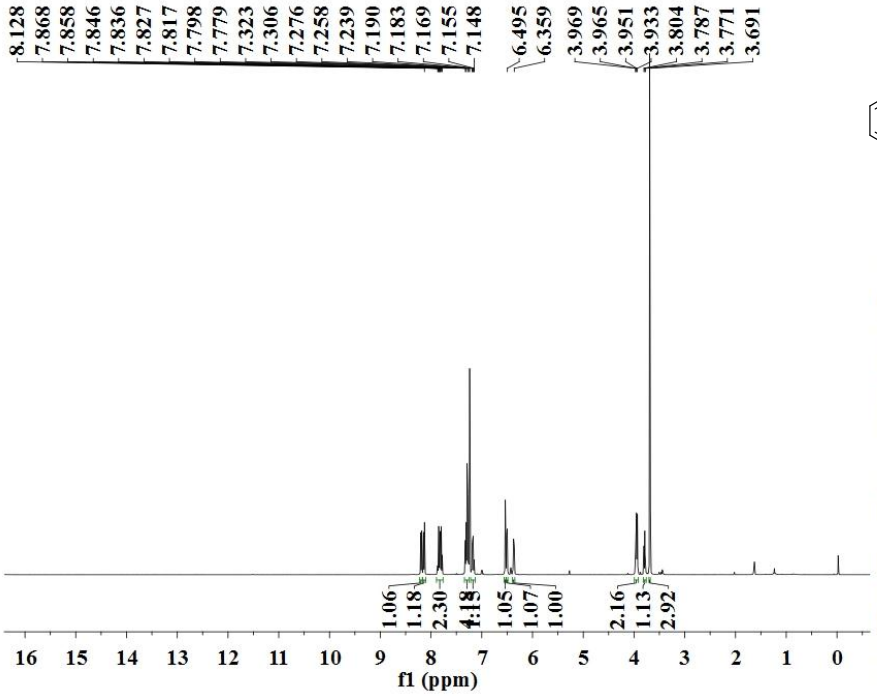


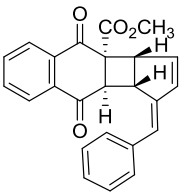
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-11-03T18:00:51
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 295.9 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -77.20
Ph1: 81.45



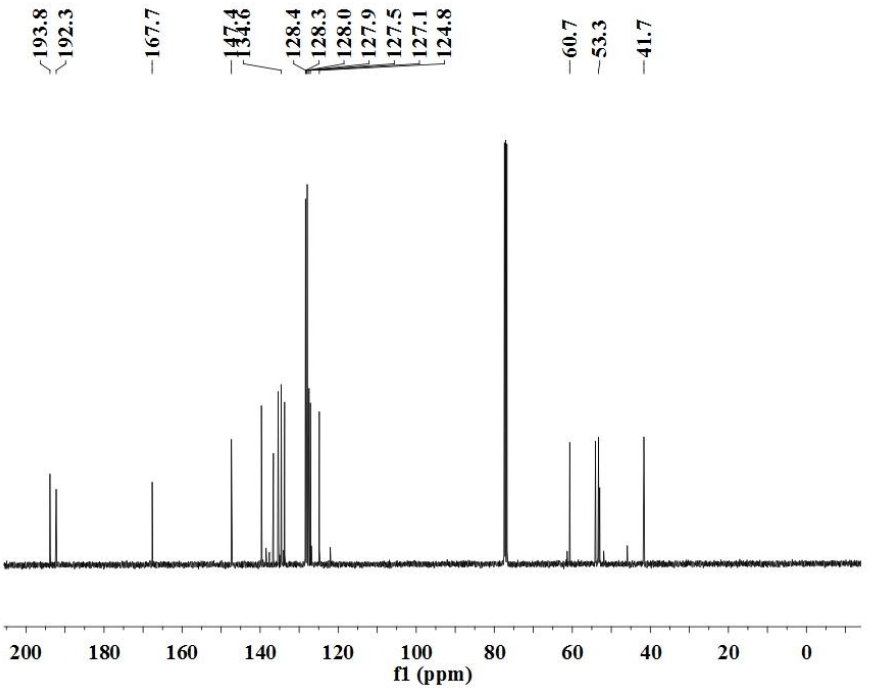


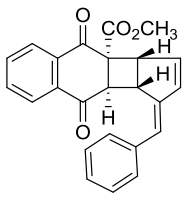
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2015-11-25T12:27:16
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 91.62
Ph1: 19.08



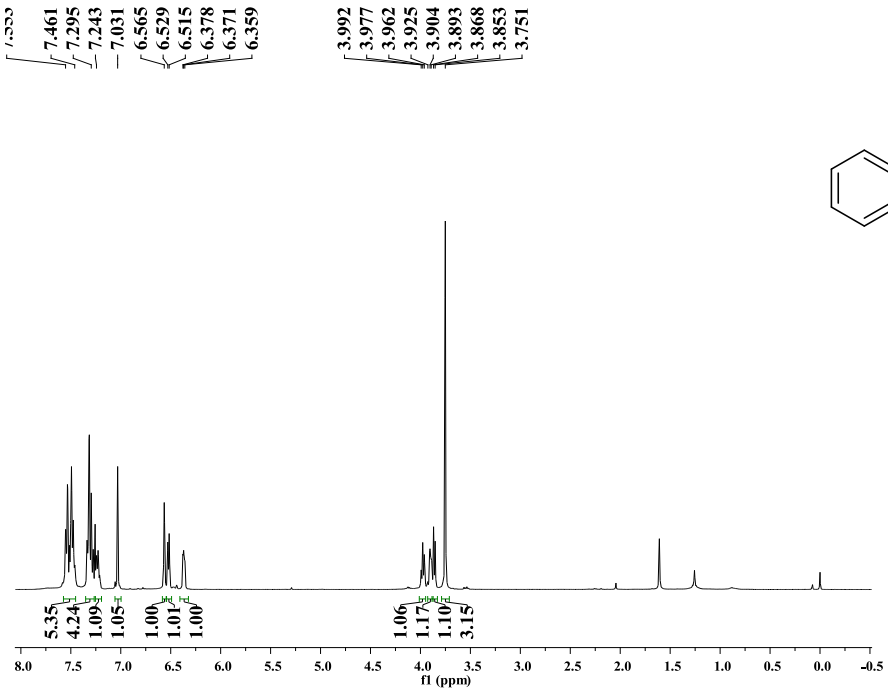


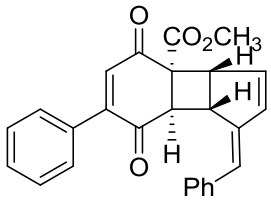
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2015-11-25T12:57:28
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 512
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -70.27
Ph1: 71.03



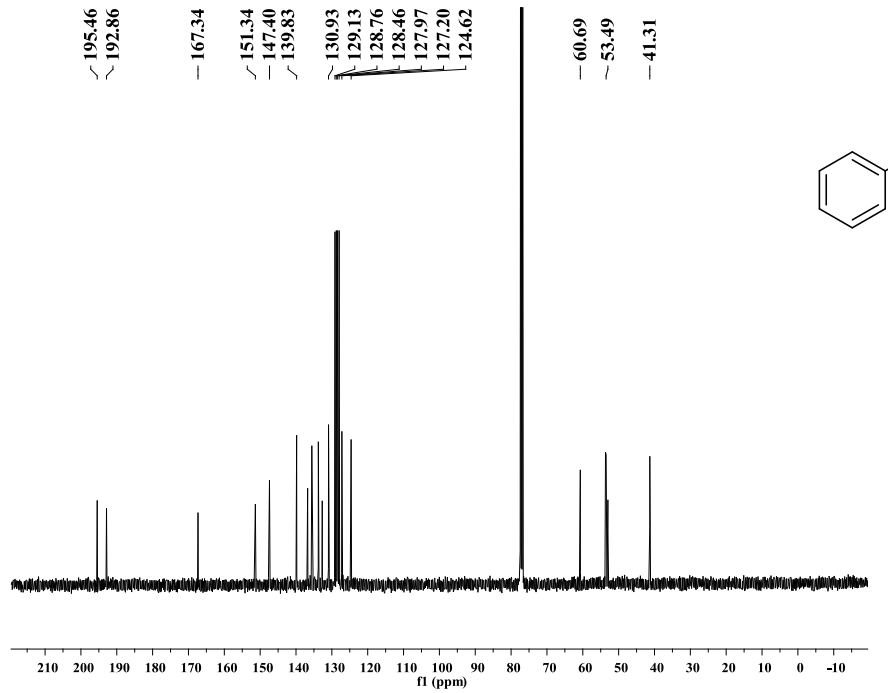


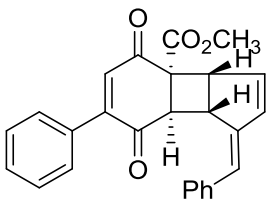
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-11-07T15:20:00
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295.4 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -264.11
Ph1: 19.20



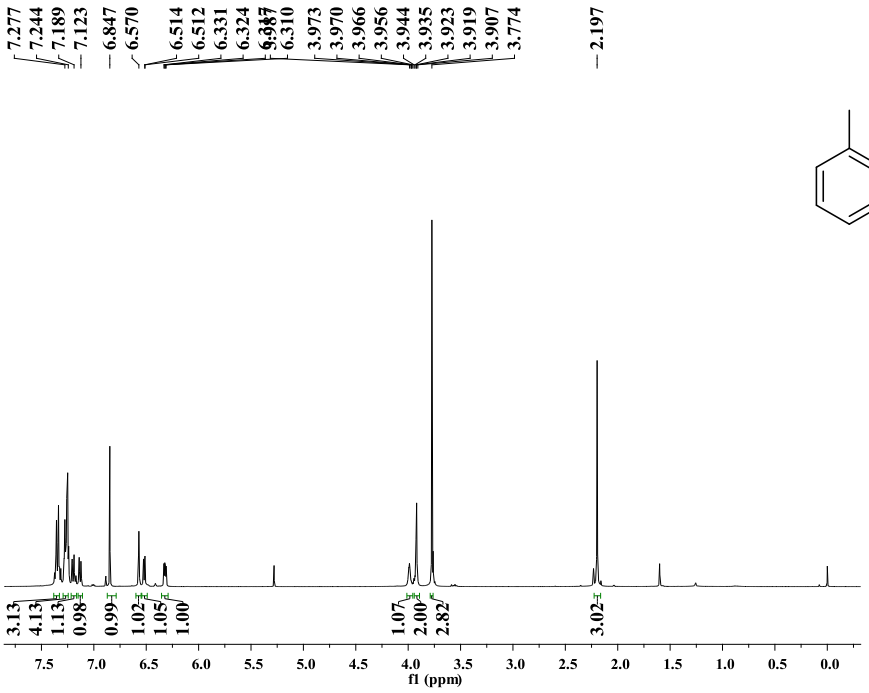


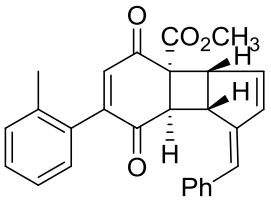
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-11-07T16:32:24
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 295.8 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -66.87
Ph1: 72.35



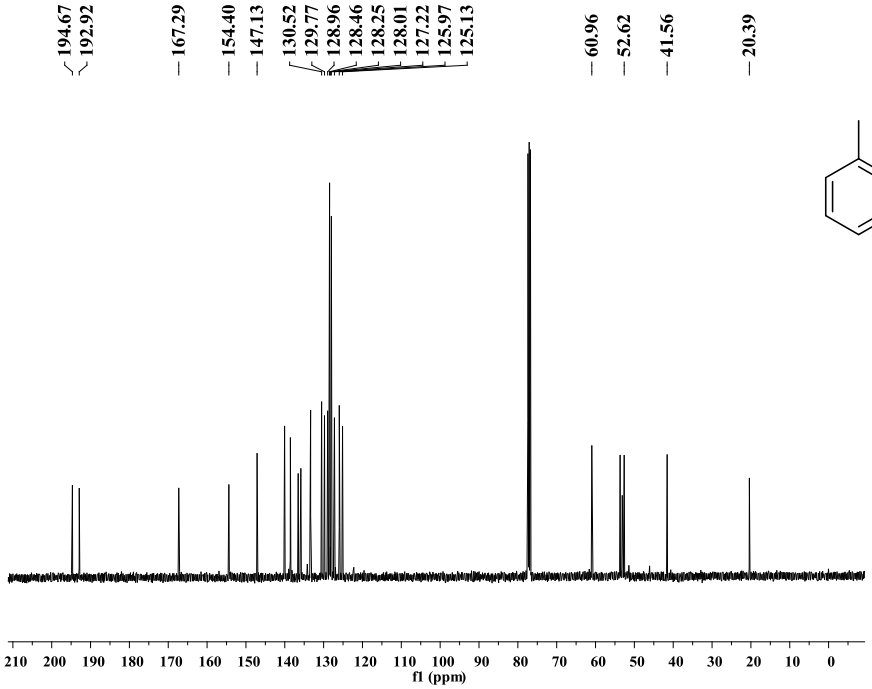


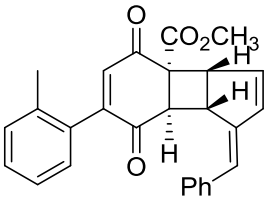
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-14T00:19:29
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.2 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 95.17
Ph1: 16.27



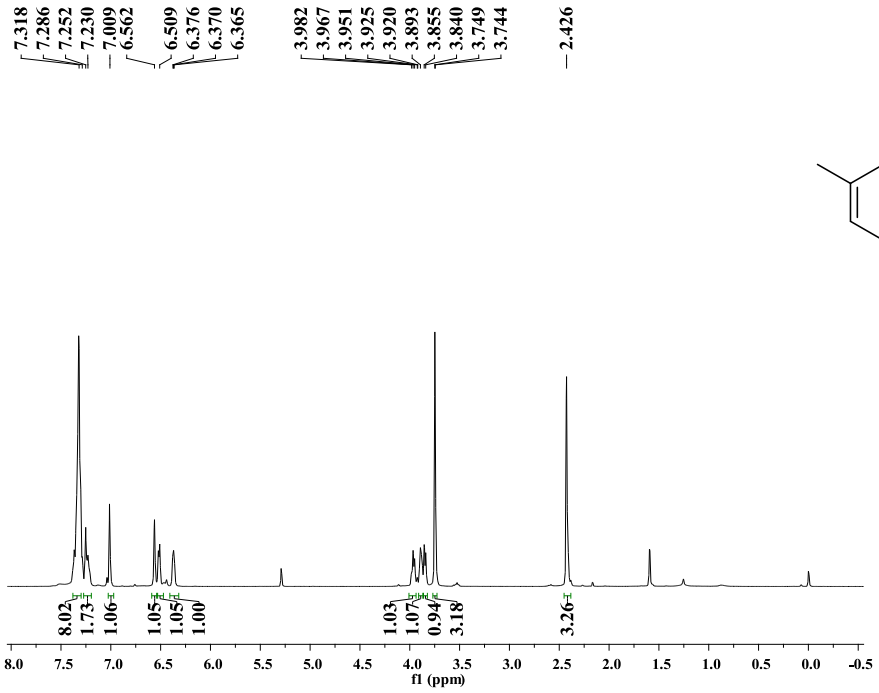


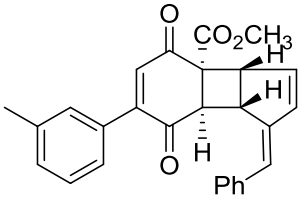
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-14T00:35:01
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.4 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -65.45
Ph1: 61.59



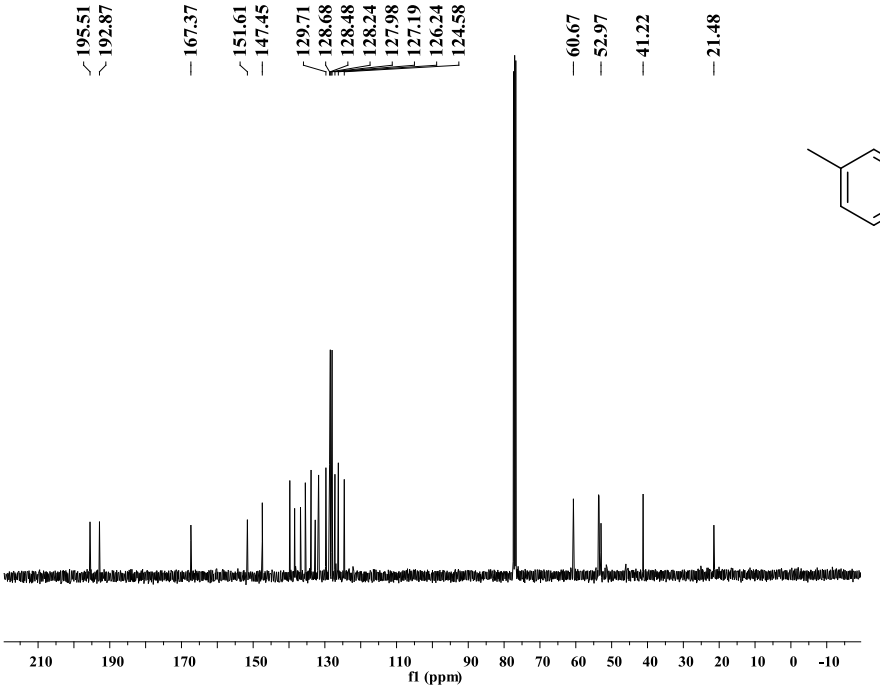


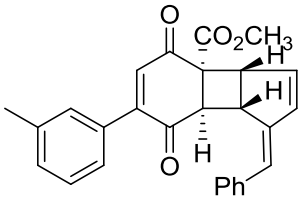
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-14T00:40:58
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 93.36
Ph1: 18.33



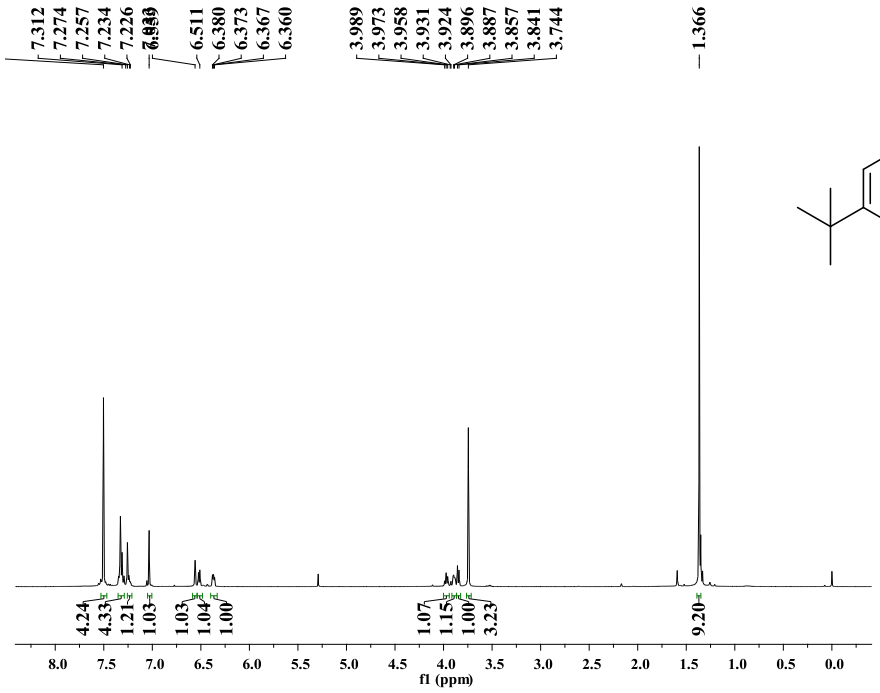


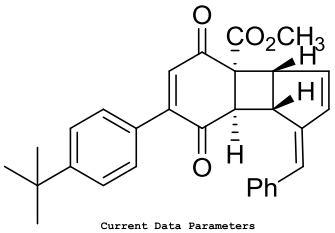
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-14T00:56:32
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.4 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -61.20
Ph1: 44.89



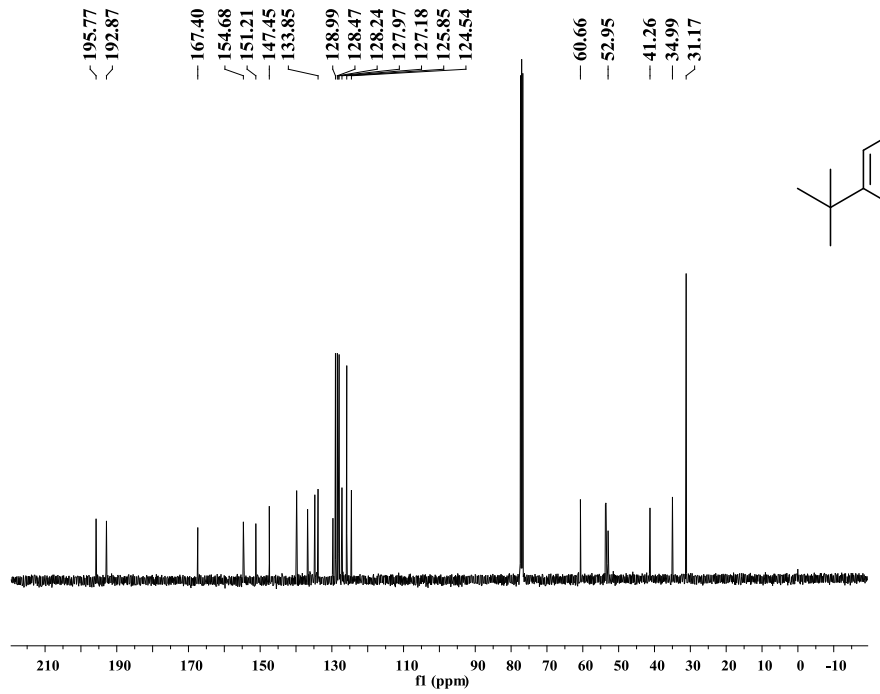


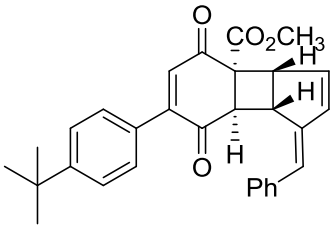
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-13T23:58:31
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.1 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 93.95
Ph1: 18.05



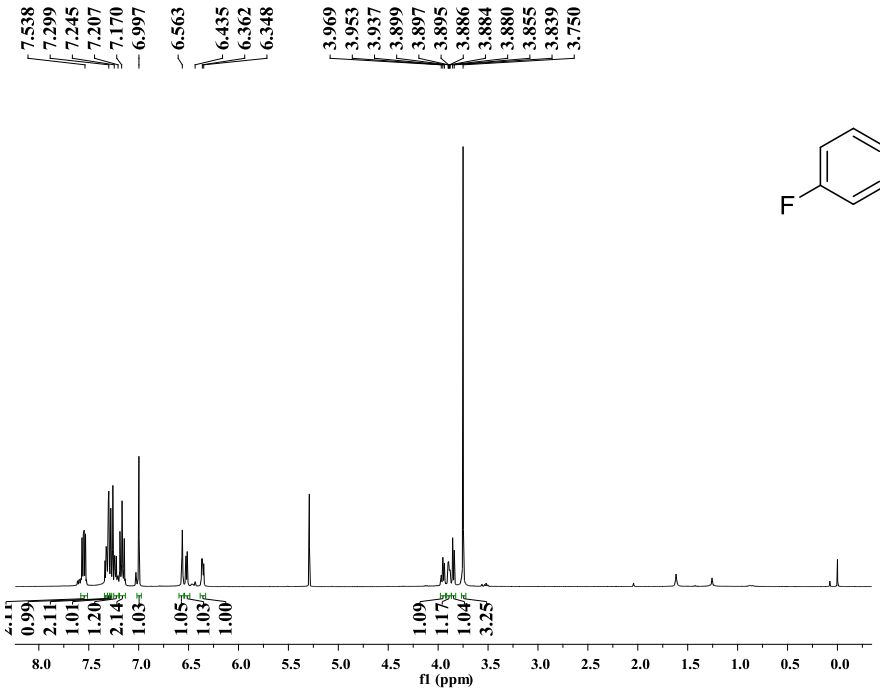


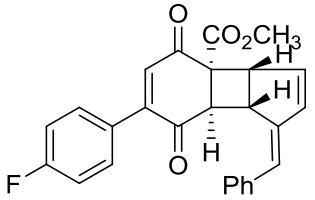
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-14T00:14:02
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.4 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -70.82
Ph1: 74.81



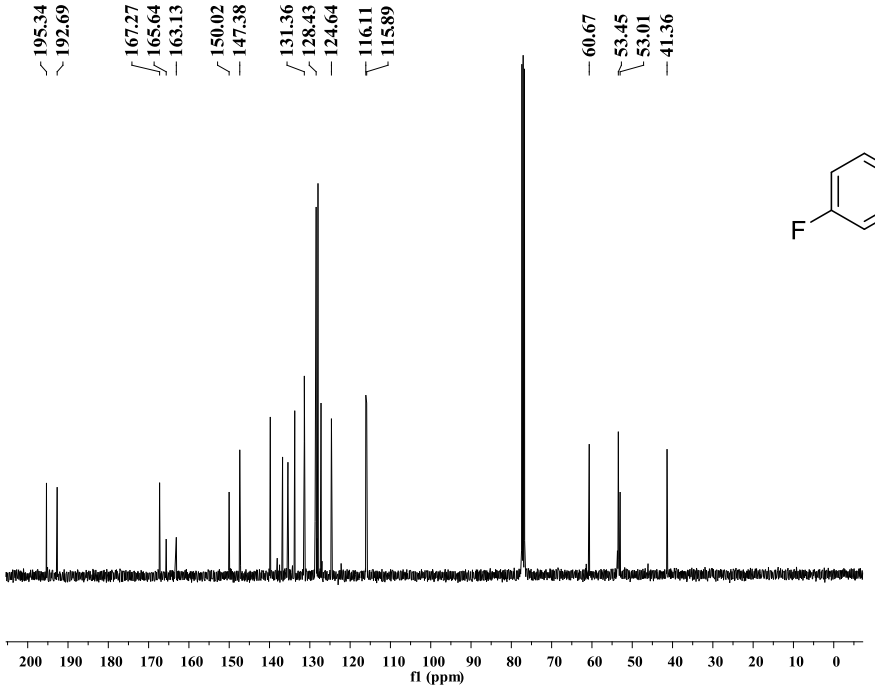


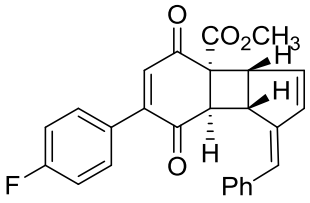
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-27T16:55:11
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.1 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 96.32
Ph1: 10.21



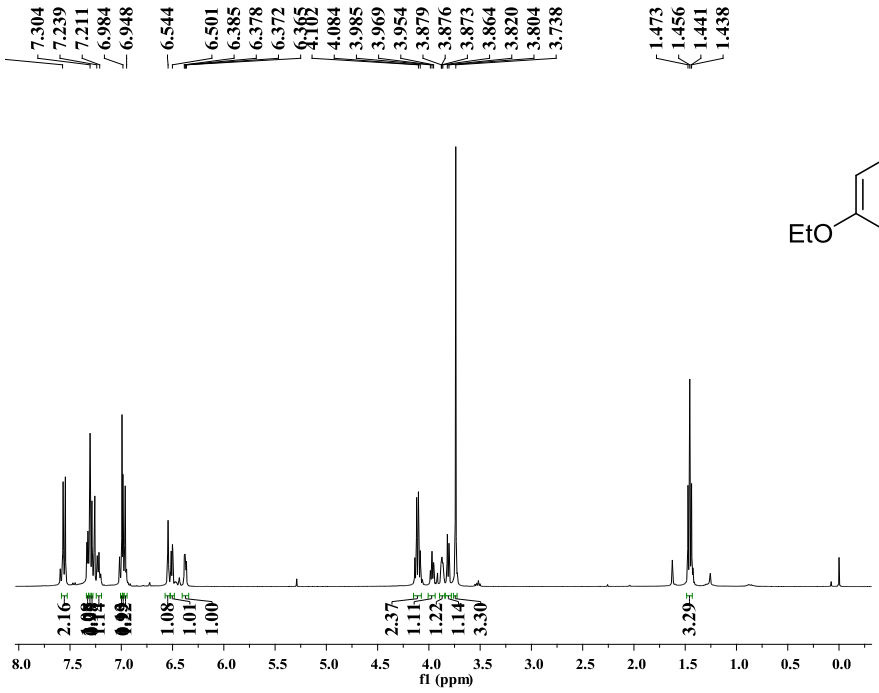


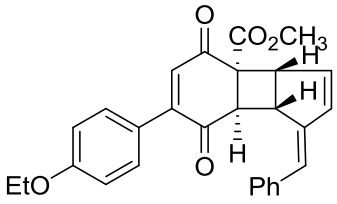
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-27T17:10:44
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.4 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -65.47
Ph1: 51.67



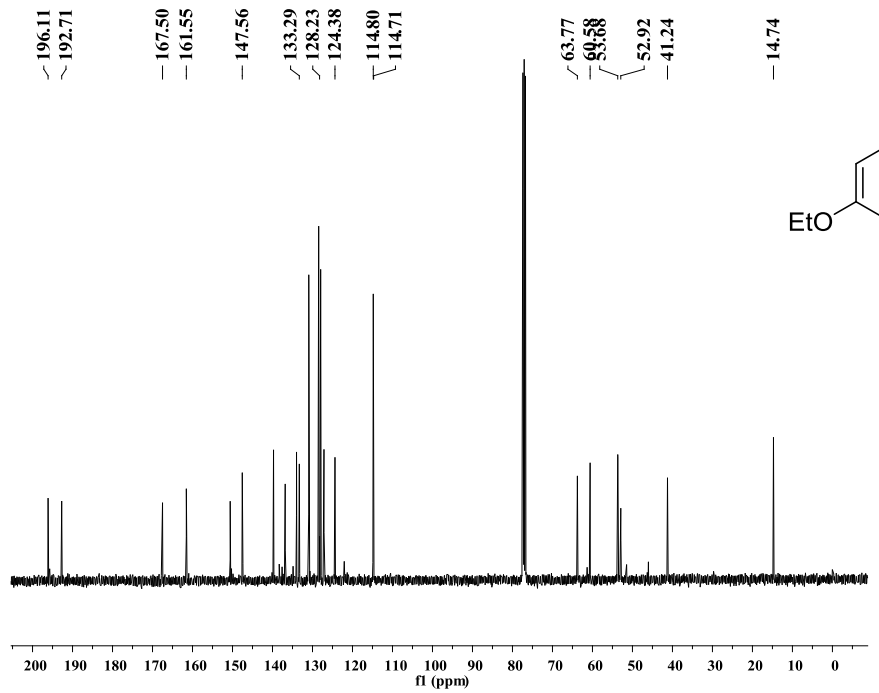


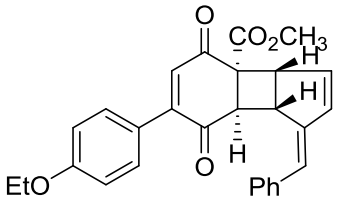
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-27T17:15:49
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.2 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 93.15
Ph1: 18.92



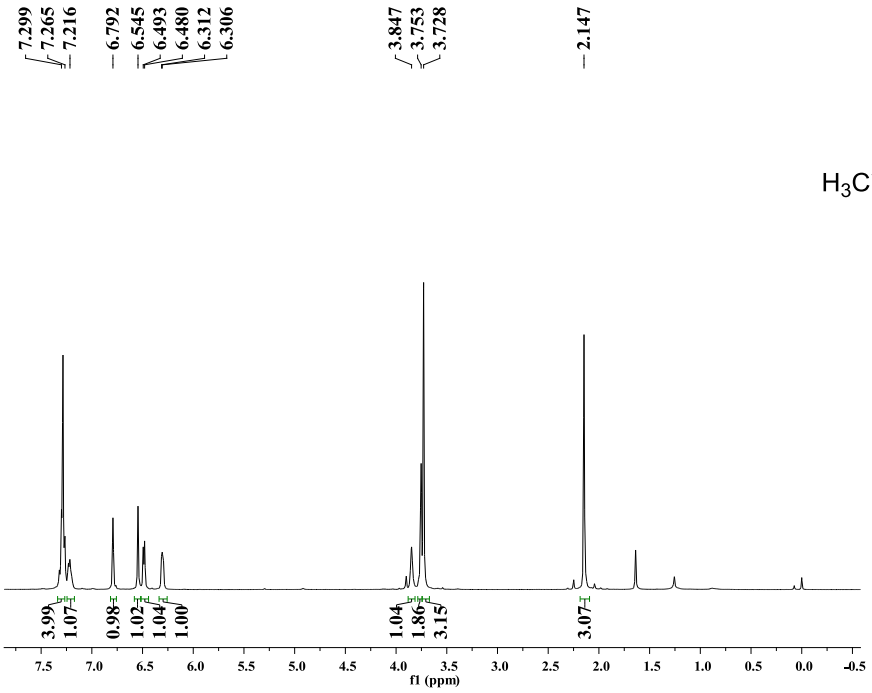


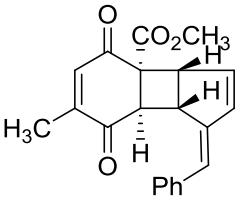
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-27T17:31:22
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.5 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -70.56
Ph1: 62.97



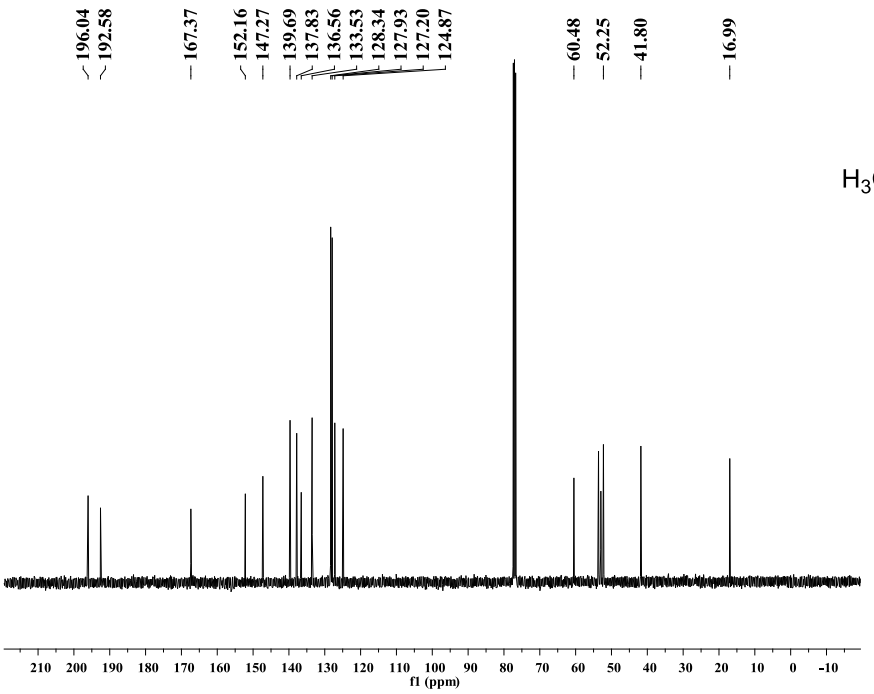


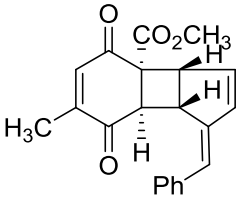
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-11-07T15:14:01
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295.5 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 97.97
Ph1: 17.15



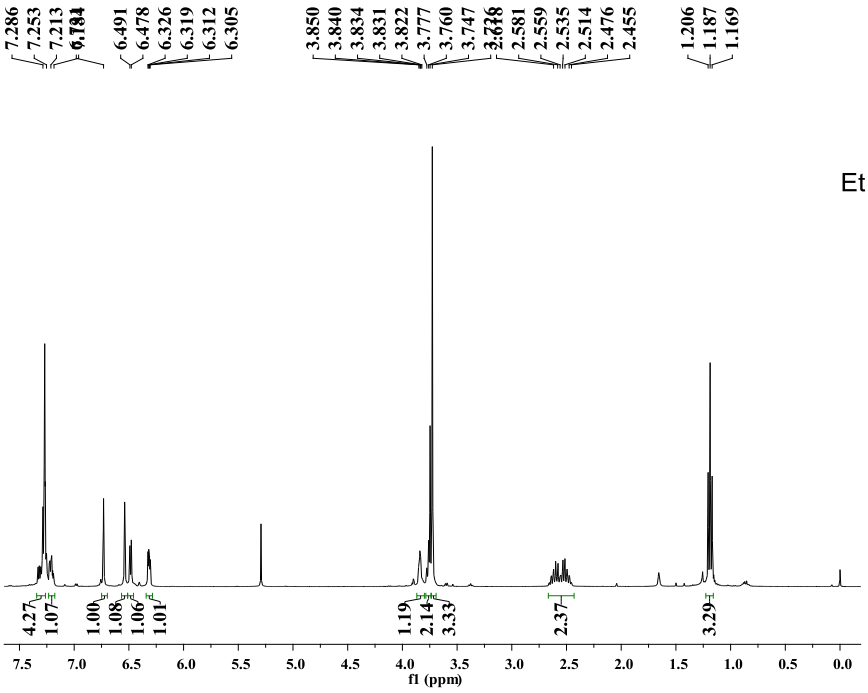


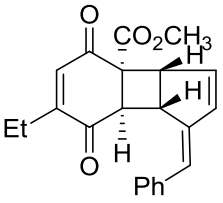
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-11-07T16:14:37
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 295.9 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -68.90
Ph1: 60.04



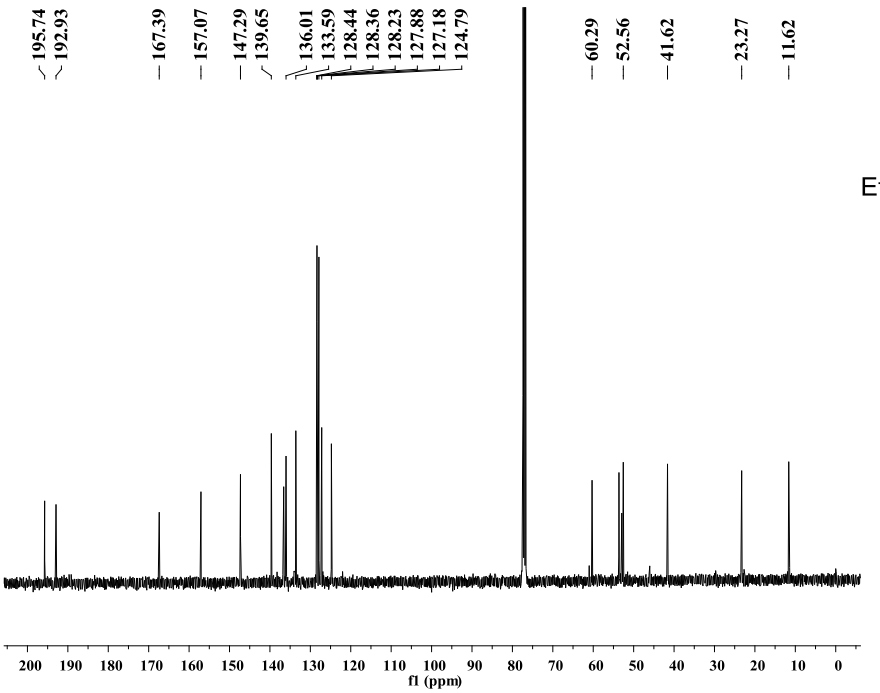


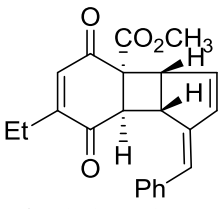
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2017-02-06T18:53:13
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295.9 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 93.16
Ph1: 18.21



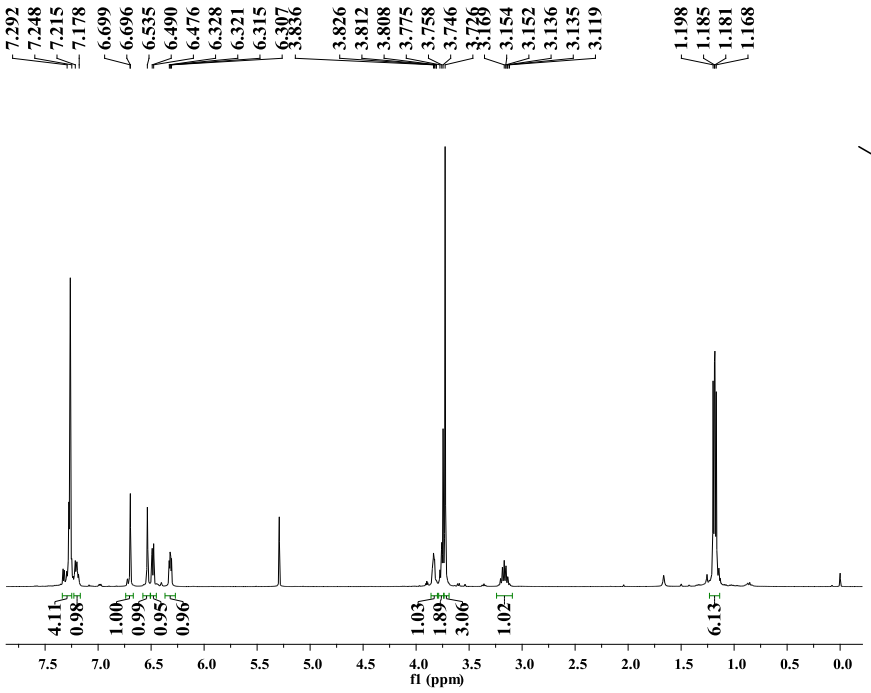


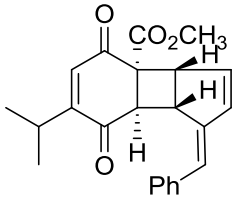
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-23T20:44:37
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 512
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.3 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -72.98
Ph1: 71.23



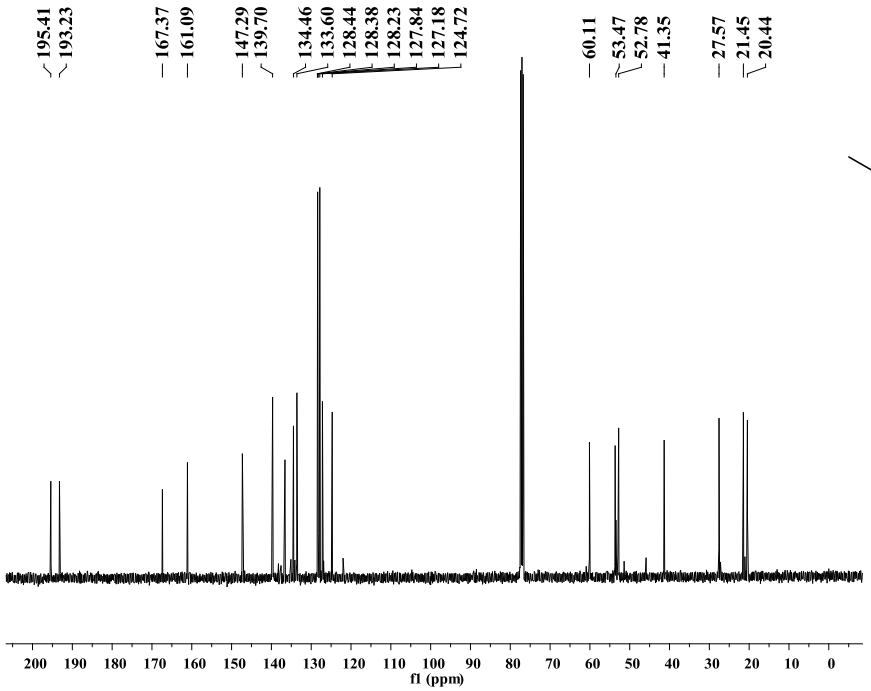


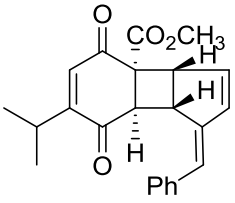
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2017-02-06T19:14:02
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295.9 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 94.30
Ph1: 18.70



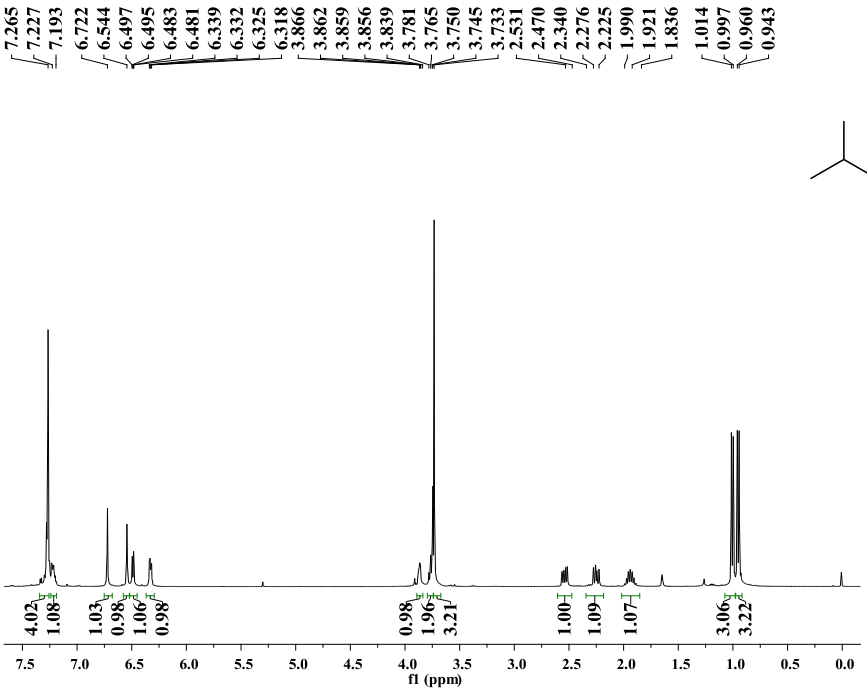


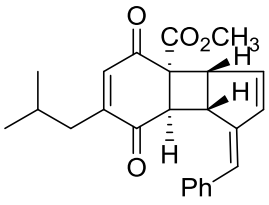
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-23T21:05:33
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.4 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -68.58
Ph1: 72.23



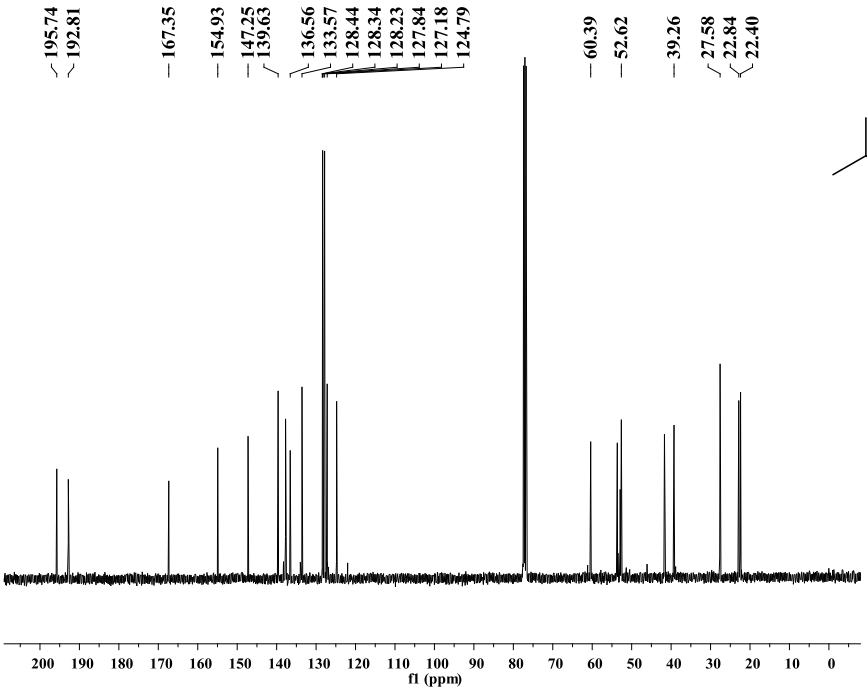


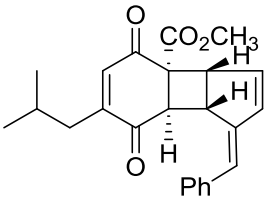
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2017-02-06T19:34:59
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295.8 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 94.75
Ph1: 16.49



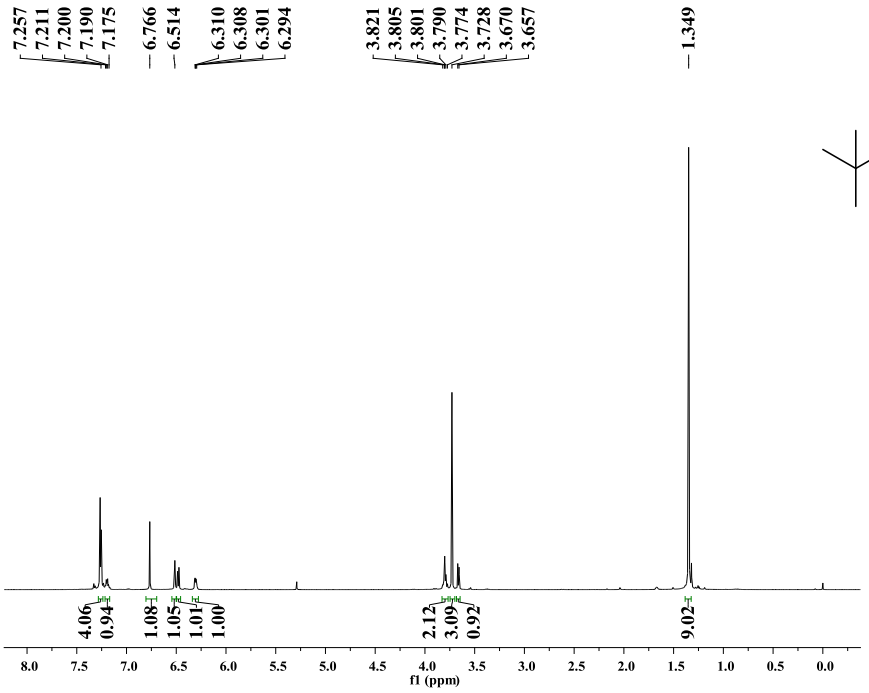


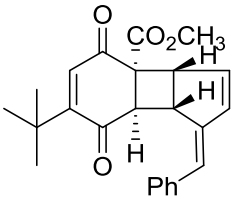
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-23T21:26:26
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.3 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -67.34
Ph1: 68.13



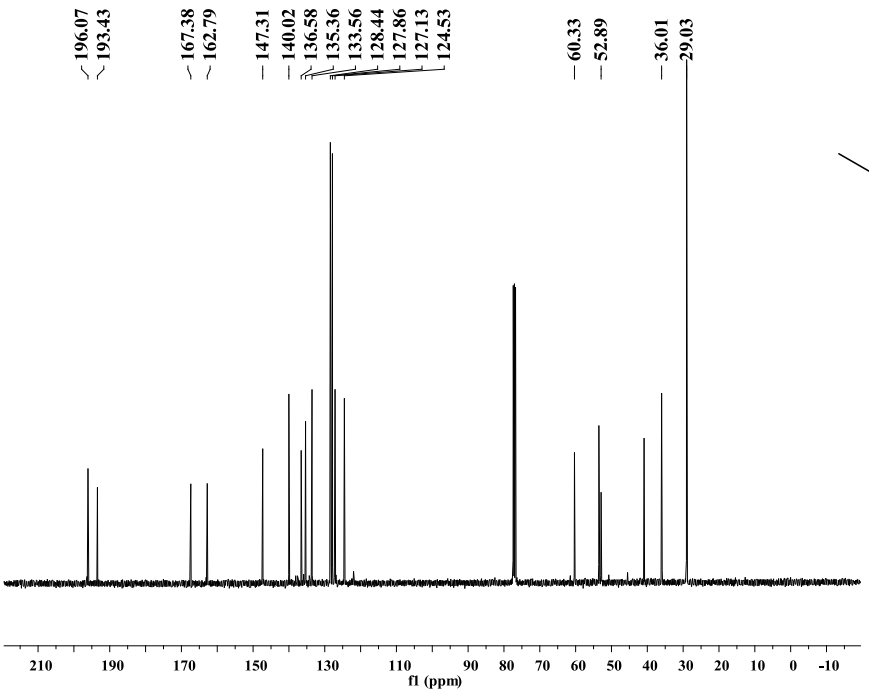


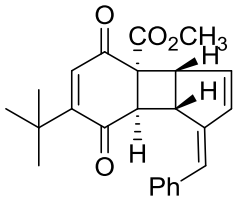
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-11-11T01:25:23
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295.2 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -259.28
Ph1: 18.61



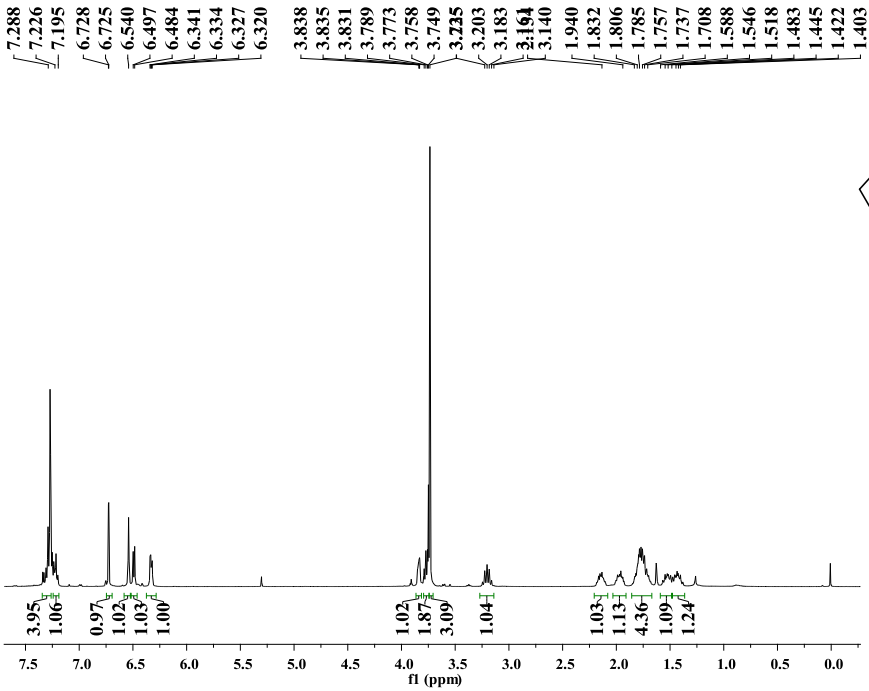


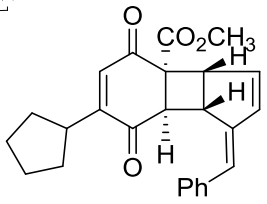
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-11-11T01:41:05
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 295.6 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -68.76
Ph1: 62.89



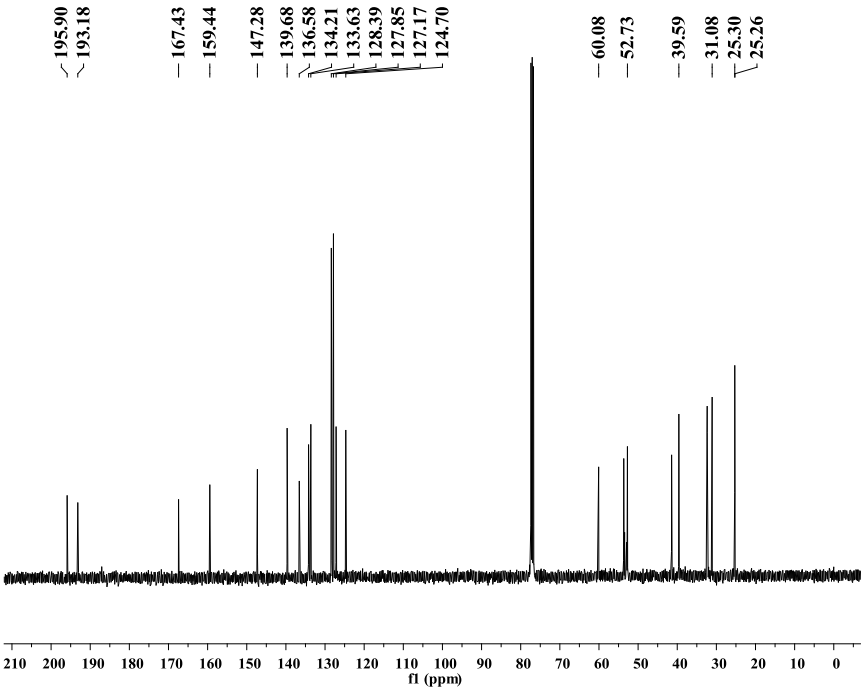


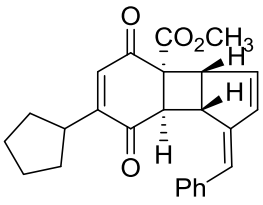
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2017-02-06T19:56:16
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 295.9 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 92.52
Ph1: 18.52



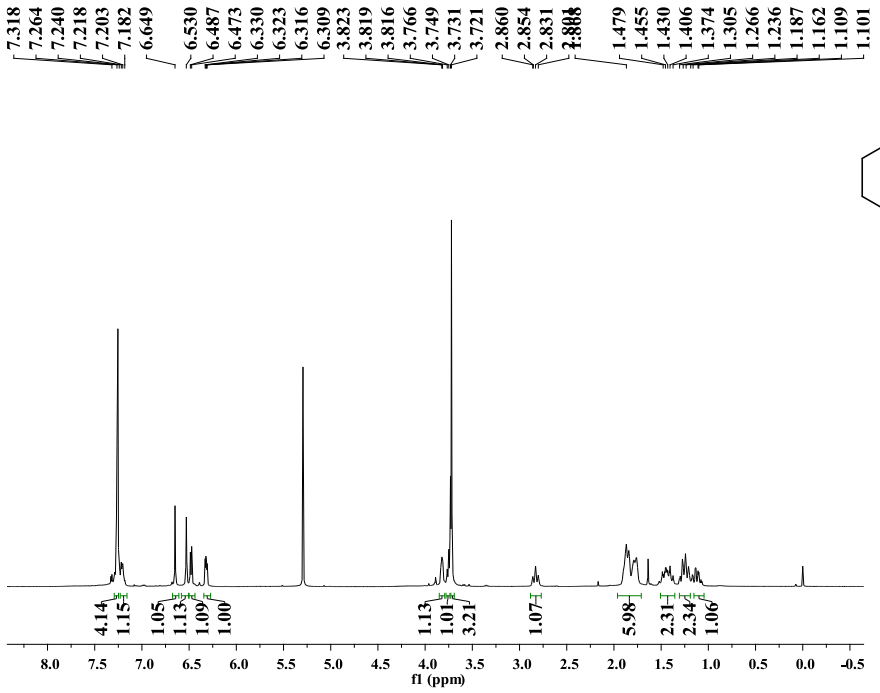


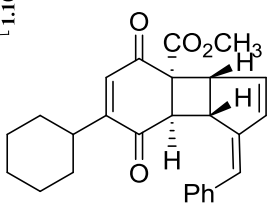
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-13T23:23:38
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.3 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -86.65
Ph1: 85.97



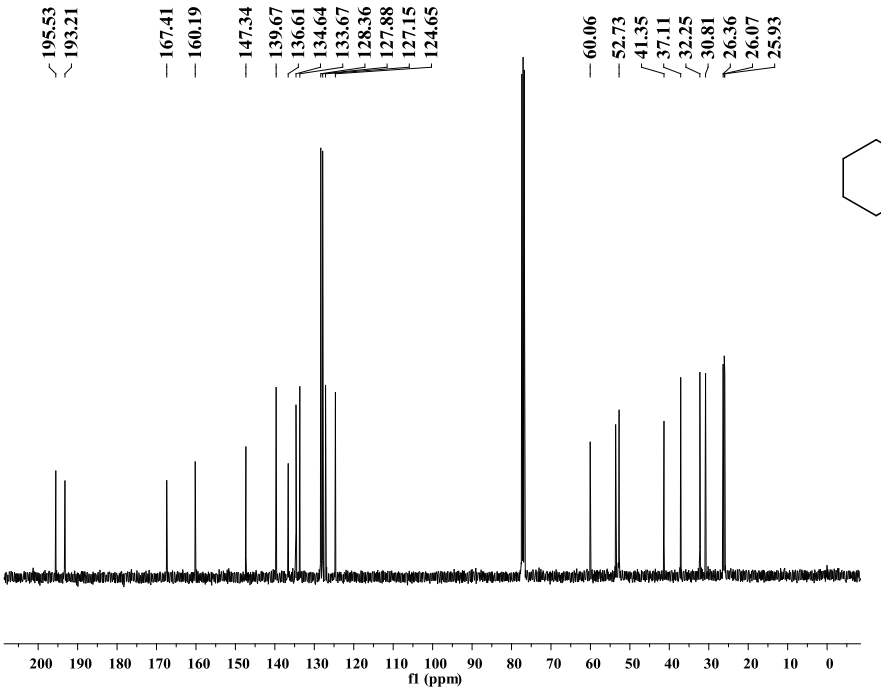


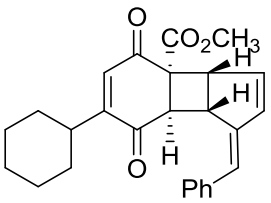
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-13T23:29:17
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.1 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 98.19
Ph1: 12.25



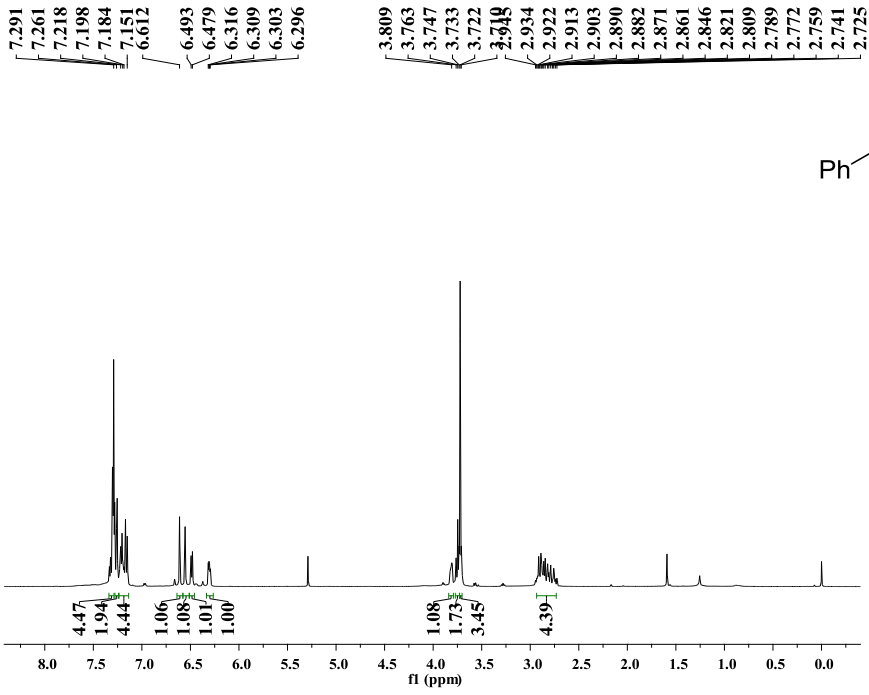


Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-13T23:44:49
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.4 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -62.73
Ph1: 54.98

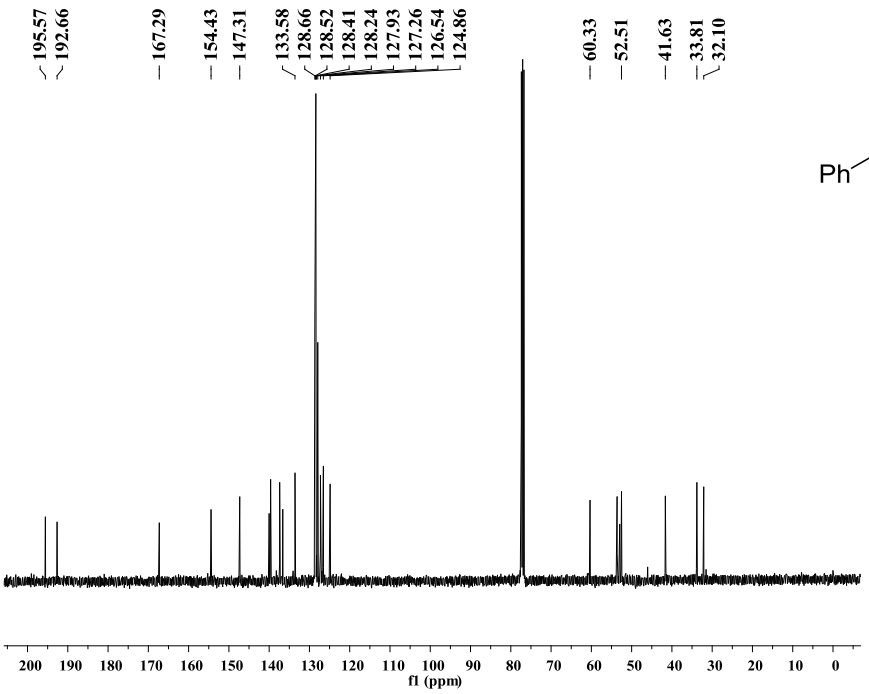


Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-23T22:31:22
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 90.37
Ph1: 21.77

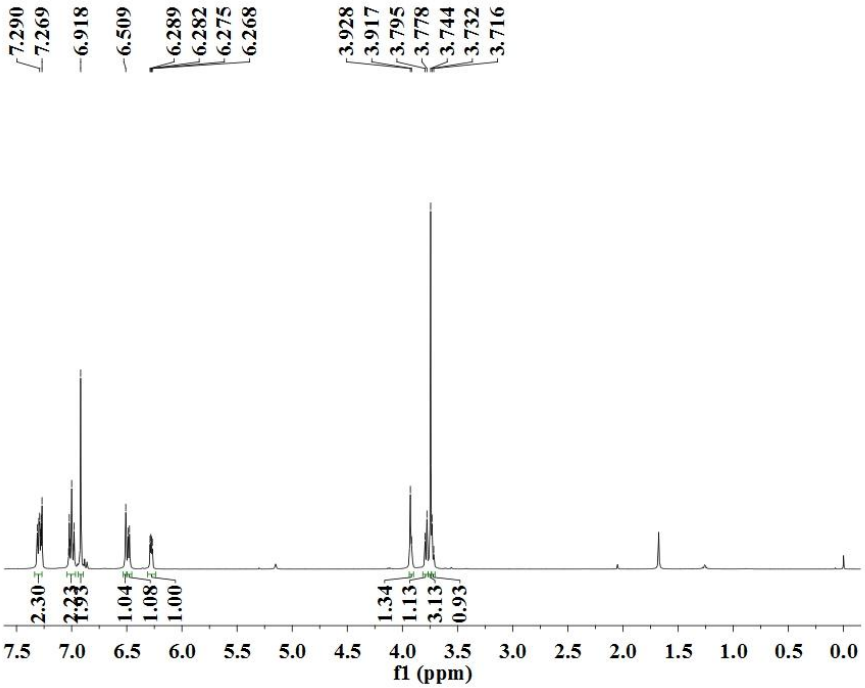


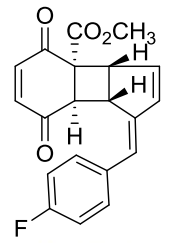
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-12-23T22:46:54
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.3 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -67.15
Ph1: 71.30



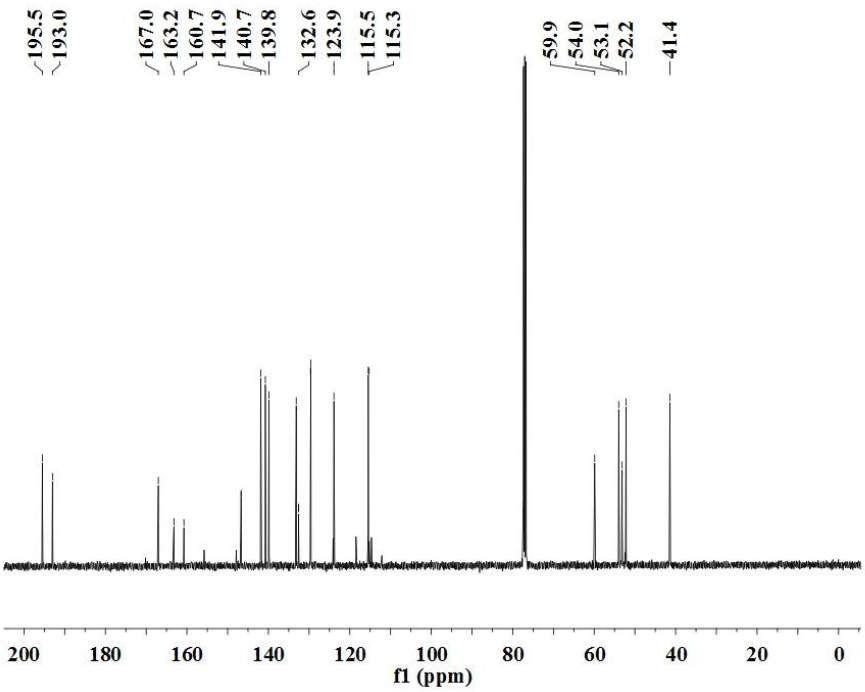


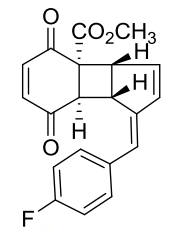
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2015-11-17T02:49:13
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 94.73
Ph1: 16.48



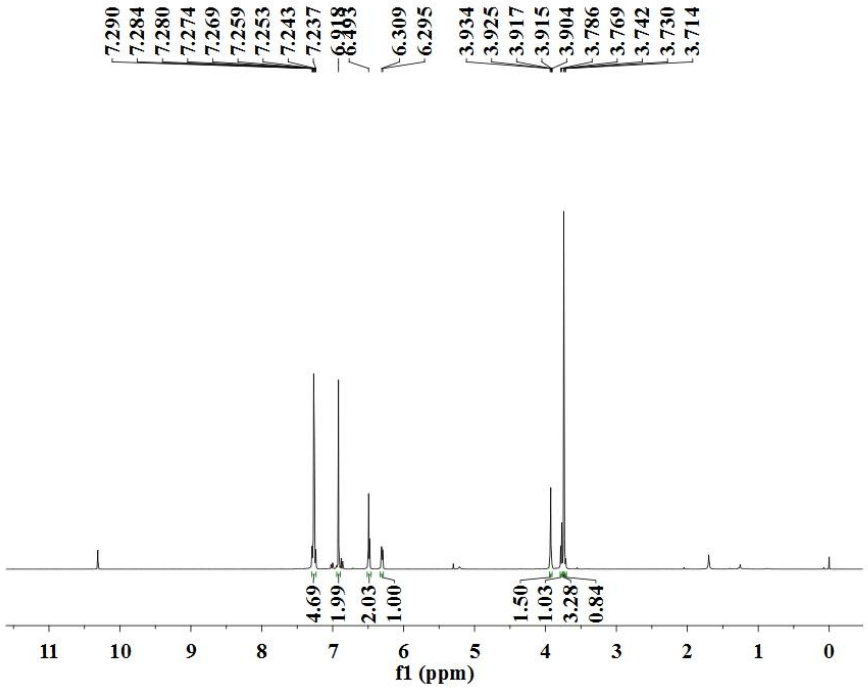


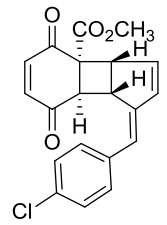
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2015-11-17T03:19:24
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 512
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -70.69
Ph1: 73.00



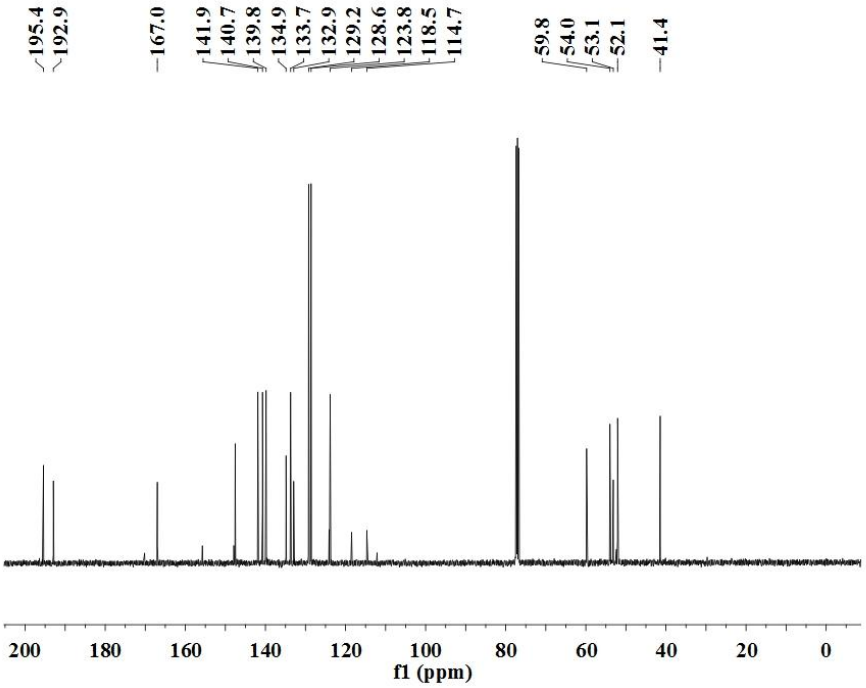


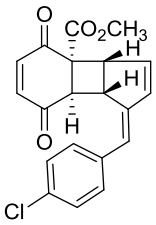
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2015-11-17T03:24:50
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 95.38
Ph1: 17.20



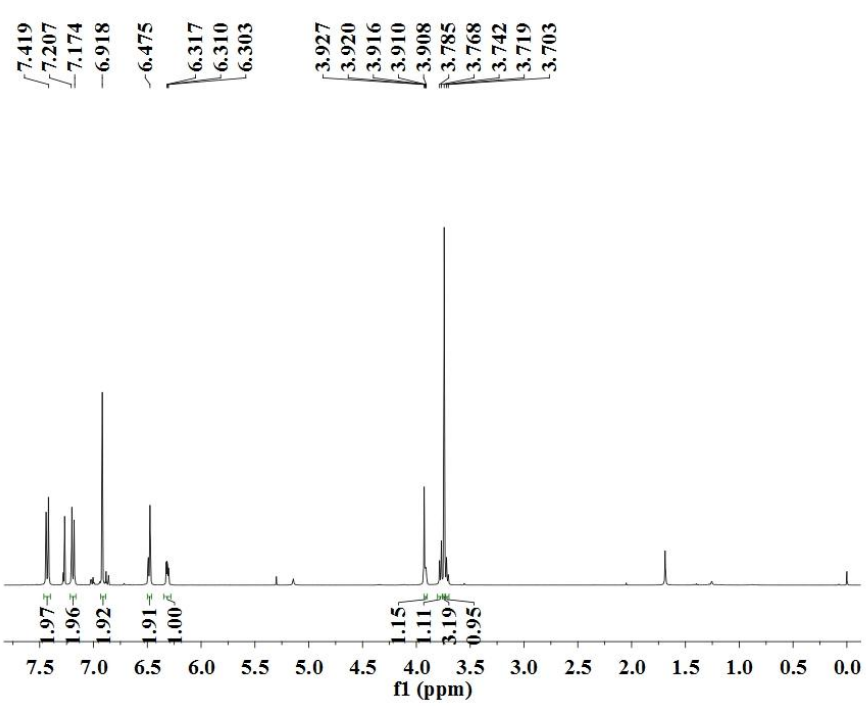


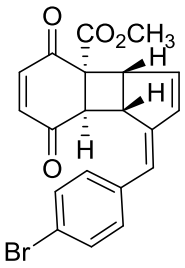
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2015-11-17T03:55:02
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 512
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -62.41
Ph1: 67.07



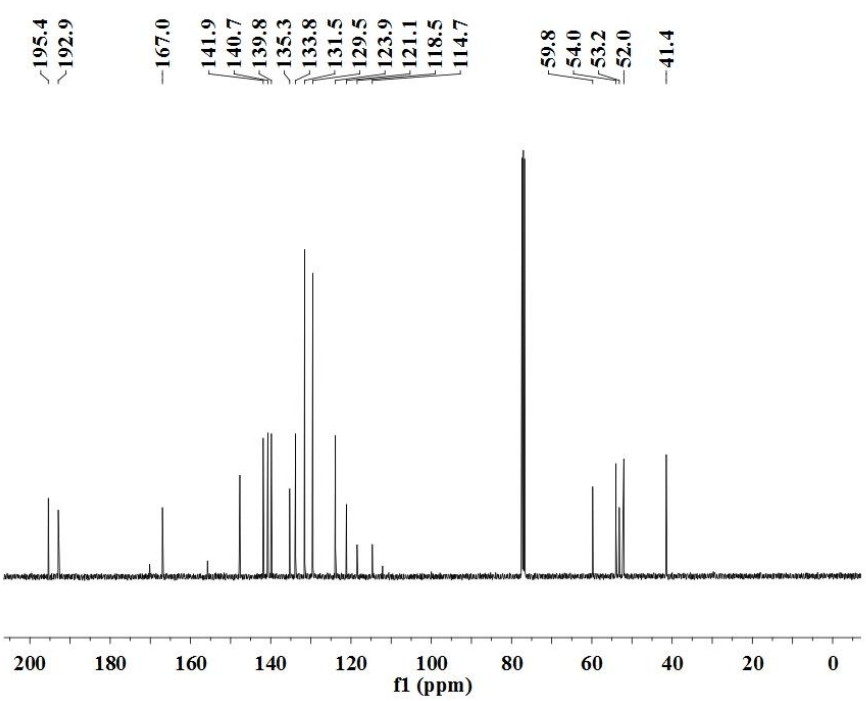


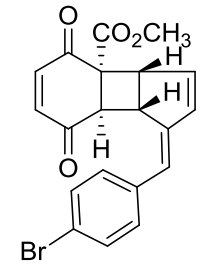
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2015-11-17T04:00:25
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -263.88
Ph1: 17.29



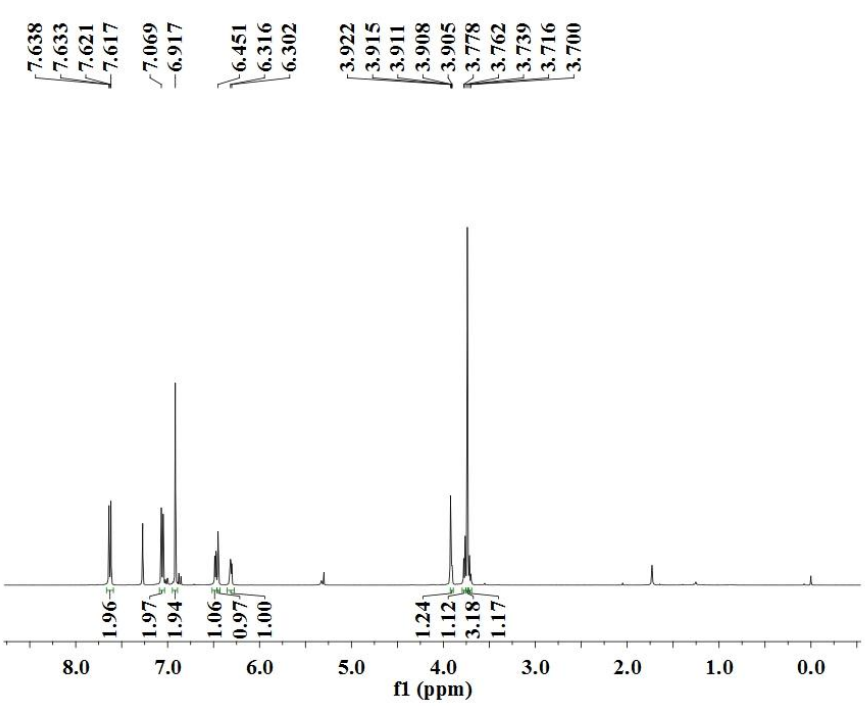


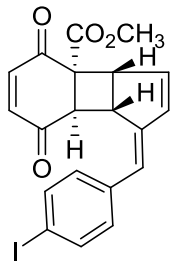
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2015-11-17T04:30:38
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 512
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -65.27
Ph1: 74.45



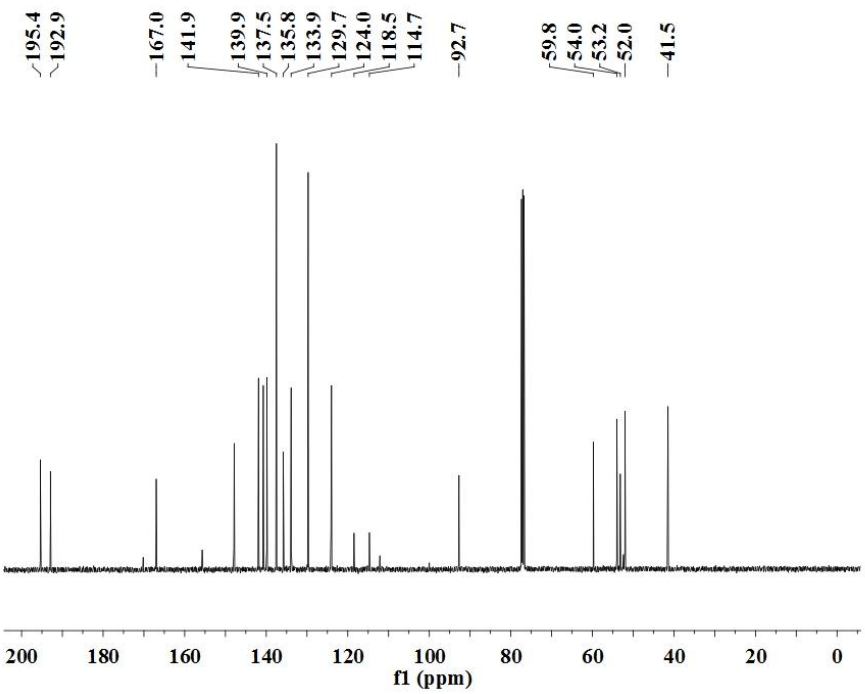


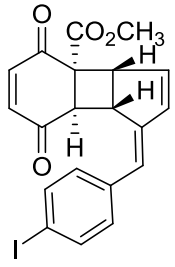
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2015-11-17T04:36:01
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 93.76
Ph1: 17.03



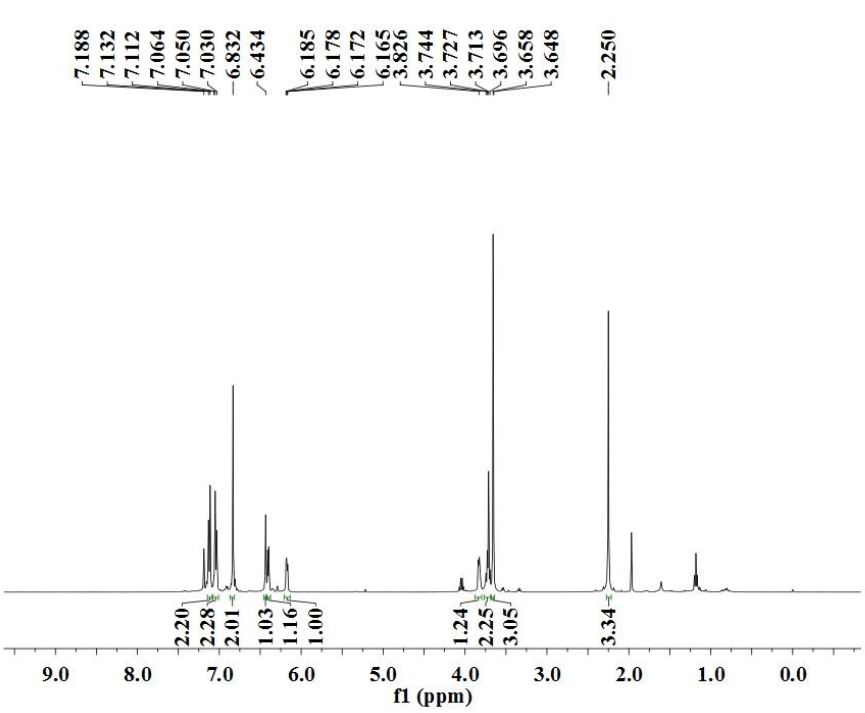


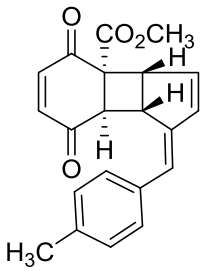
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2015-11-17T05:06:14
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 512
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -67.77
Ph1: 62.22



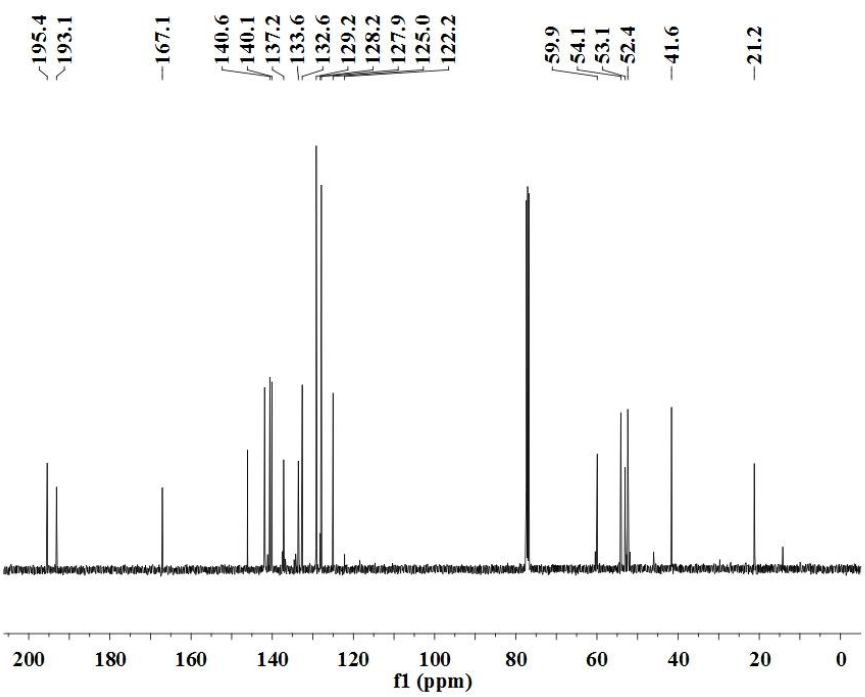


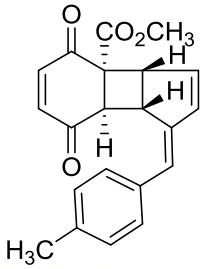
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-03-03T11:51:54
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 95.10
Ph1: 15.73



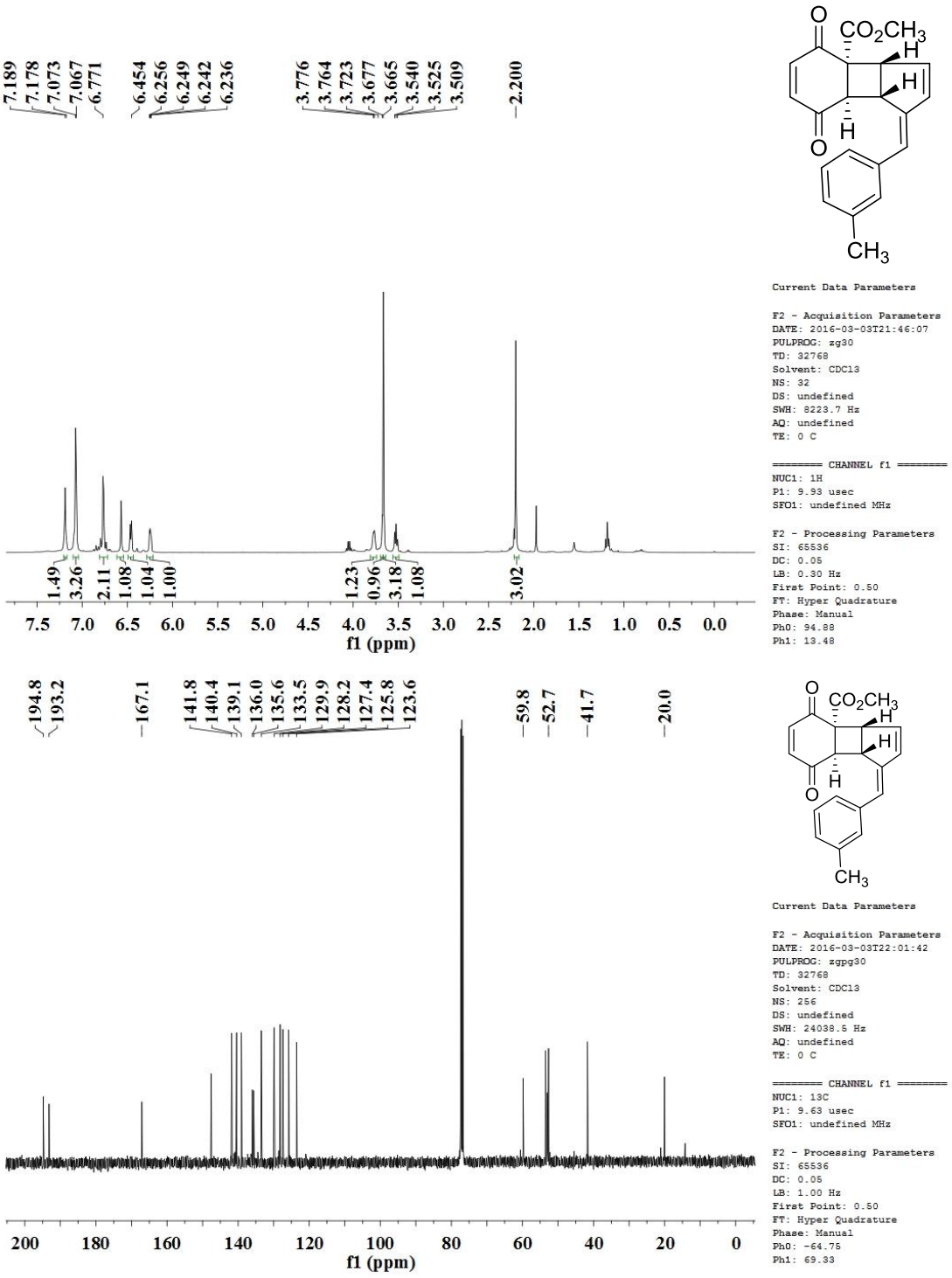


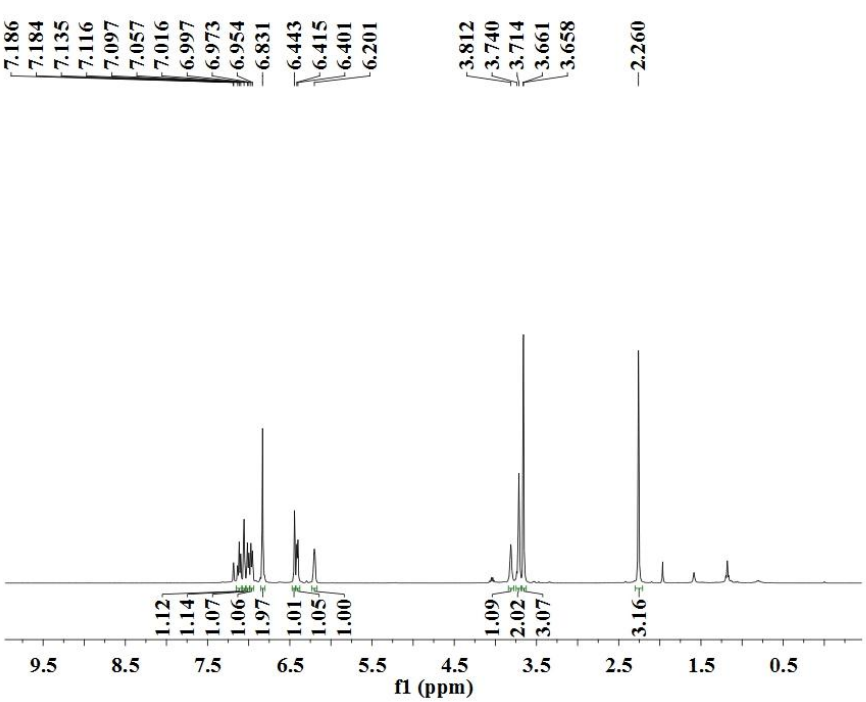
Current Data Parameters

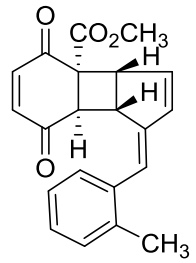
F2 - Acquisition Parameters
DATE: 2016-03-03T12:07:29
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -62.03
Ph1: 53.60


7.189
7.178
7.073
7.067
6.771
6.454
6.256
6.249
6.242
6.236
3.776
3.764
3.723
3.677
3.665
3.540
3.525
3.509
2.200
O
CO2CH3
H
H
H
O
CH3
1.49
3.26
2.11
1.08
1.04
1.00
1.23
0.96
3.18
1.08
3.02
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2016-03-03T21:46:07
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 94.88
Ph1: 13.48
194.8
193.2
167.1
141.8
140.4
139.1
136.0
135.6
133.5
129.9
128.2
127.4
125.8
123.6
59.8
52.7
41.7
20.0
O
CO2CH3
H
H
H
O
CH3
200 180 160 140 120 100 80 60 40 20 0
f1 (ppm)
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2016-03-03T22:01:42
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -64.75
Ph1: 69.33



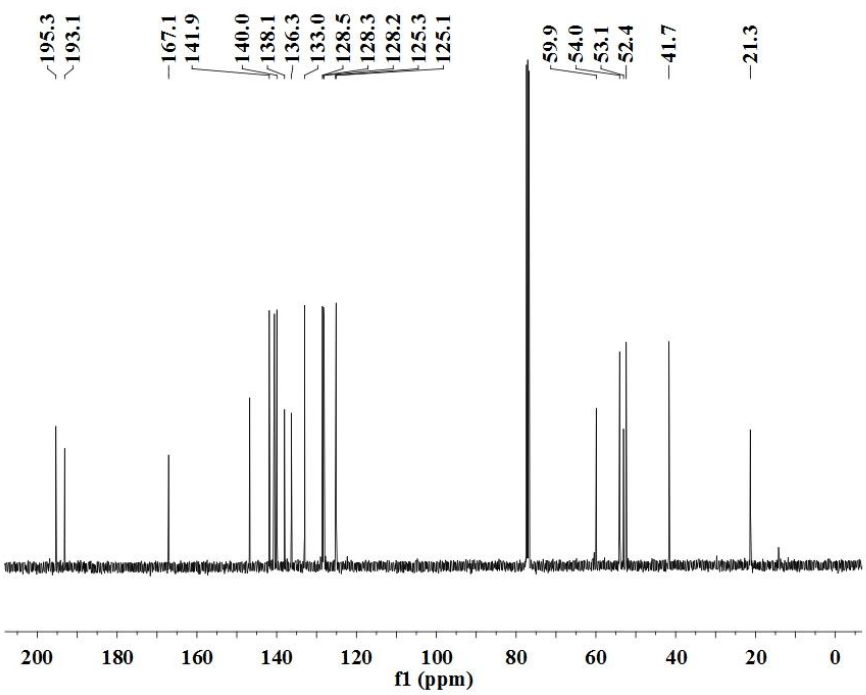


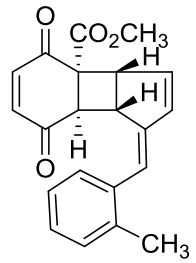
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-03-04T11:22:36
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 93.76
Ph1: 17.64



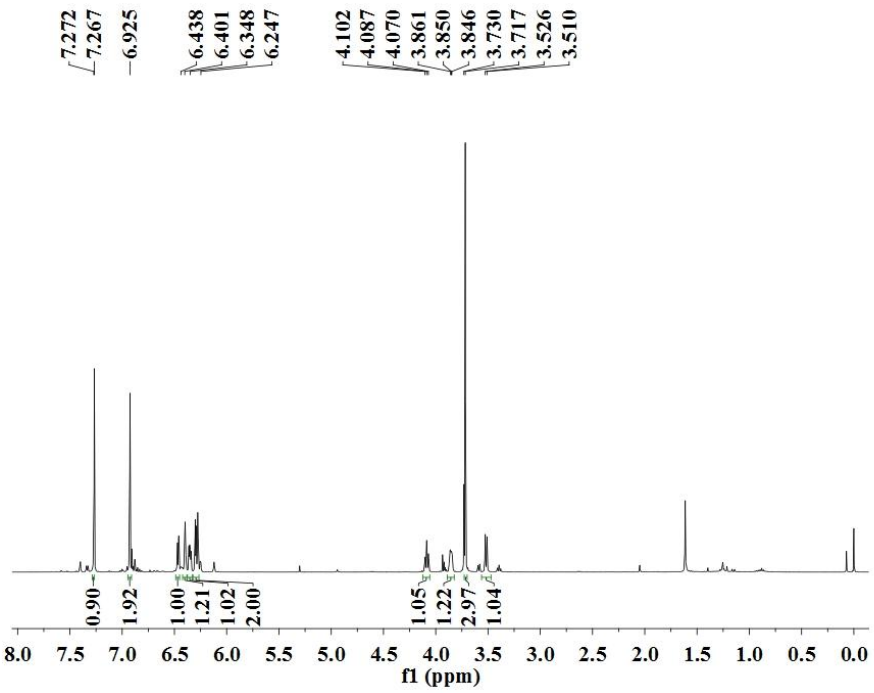


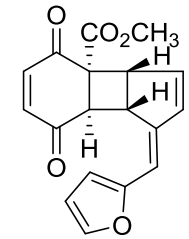
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-03-04T11:38:11
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -66.68
Ph1: 64.39



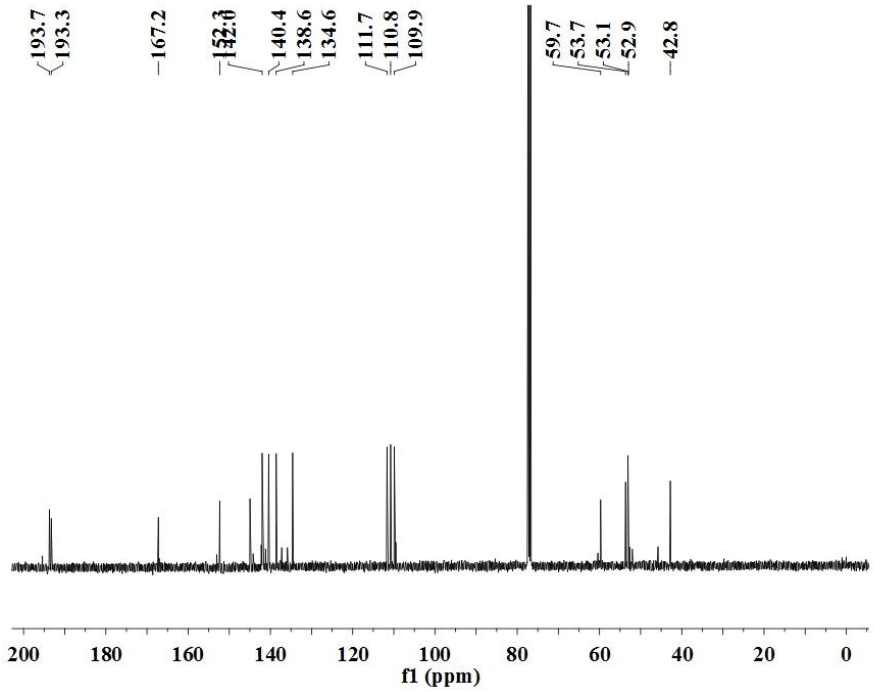


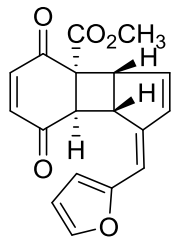
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2015-12-03T12:12:51
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 95.26
Ph1: 13.75



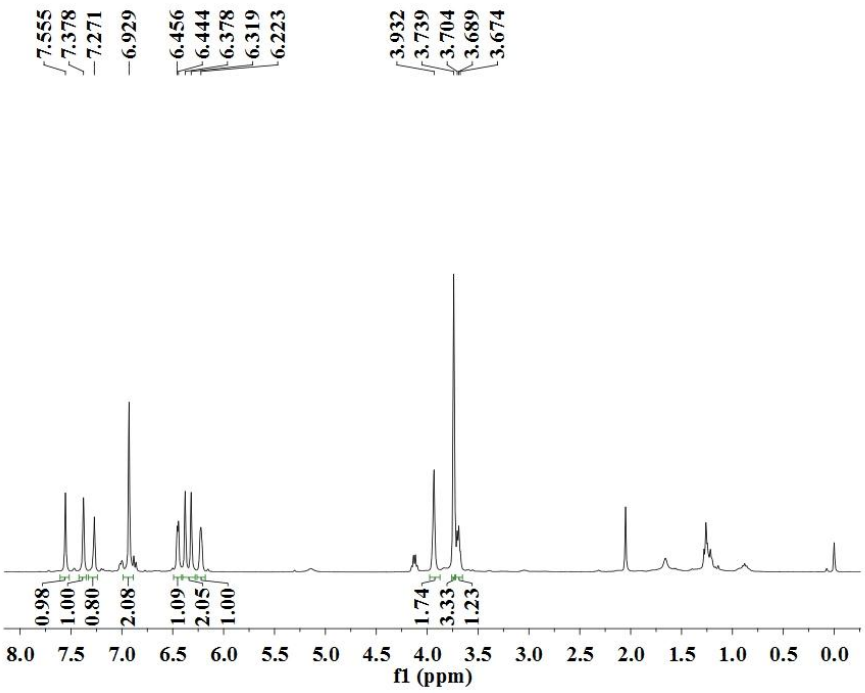


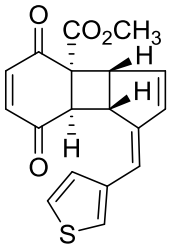
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2015-12-03T12:43:03
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 512
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -85.45
Ph1: 91.07



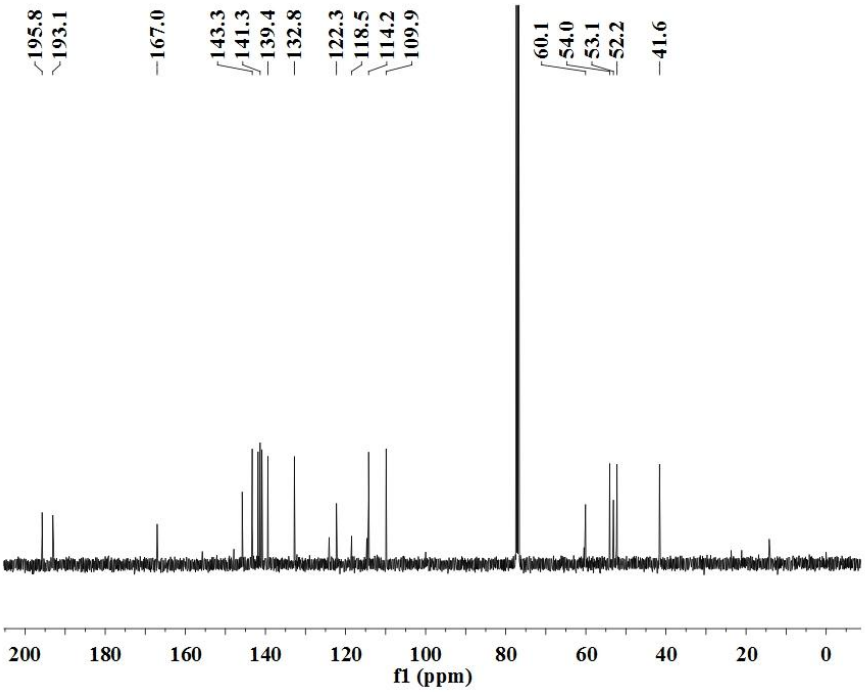


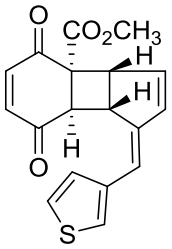
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-03-11T12:38:46
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 96.47
Ph1: 13.57



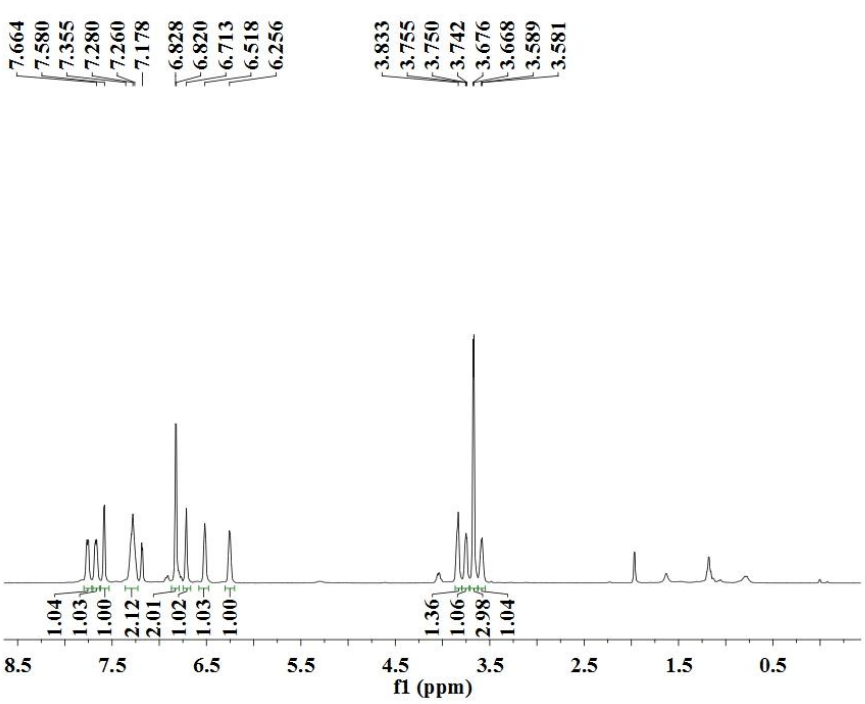


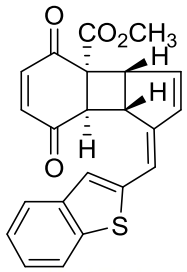
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-03-11T12:54:20
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -64.50
Ph1: 68.89



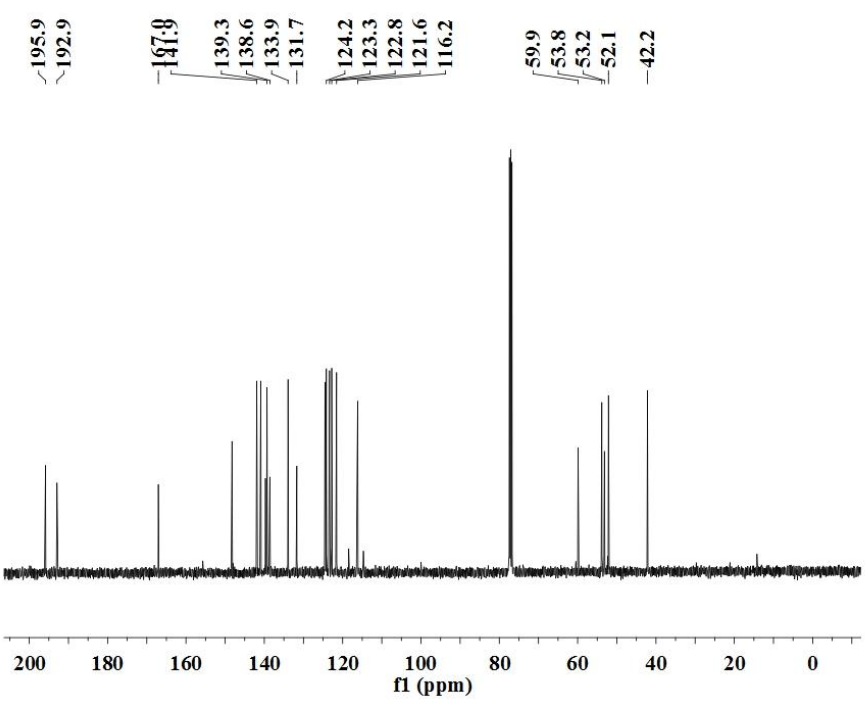


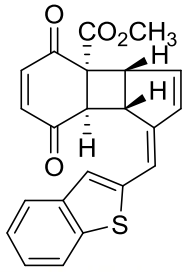
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-03-08T11:56:45
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 95.97
Ph1: 14.46



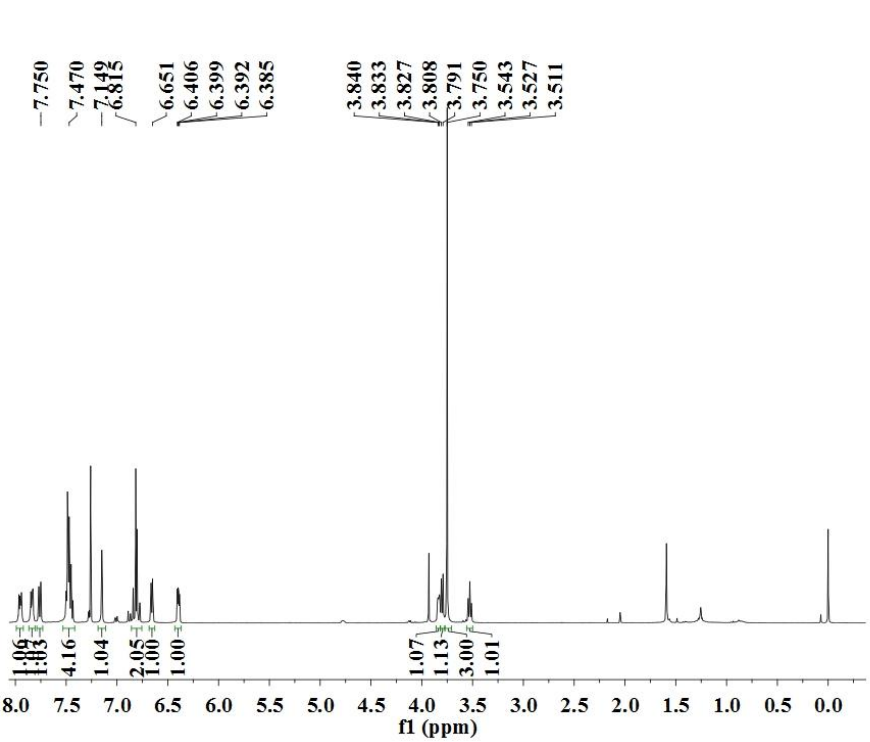


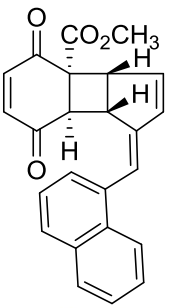
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-03-08T12:12:20
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -65.48
Ph1: 56.08



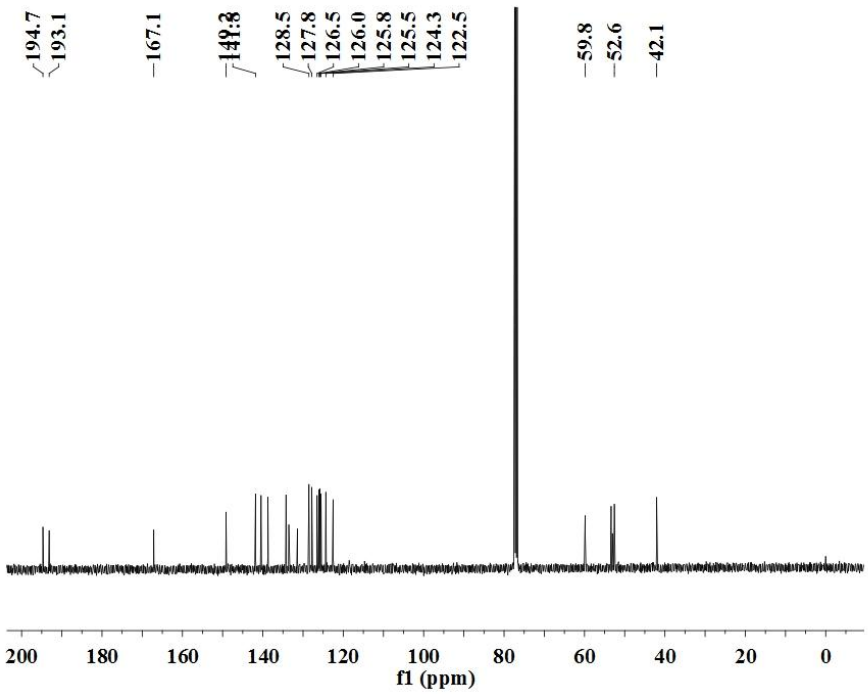


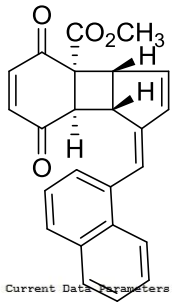
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-04-19T10:22:38
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 95.78
Ph1: 11.24



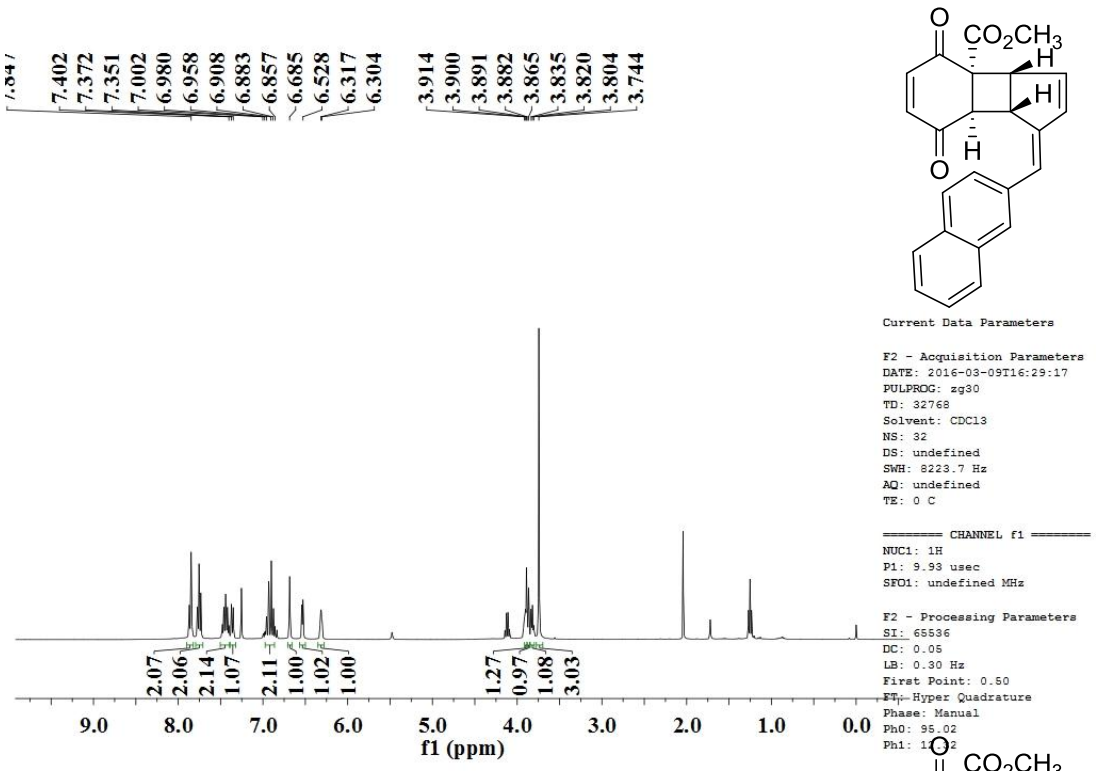


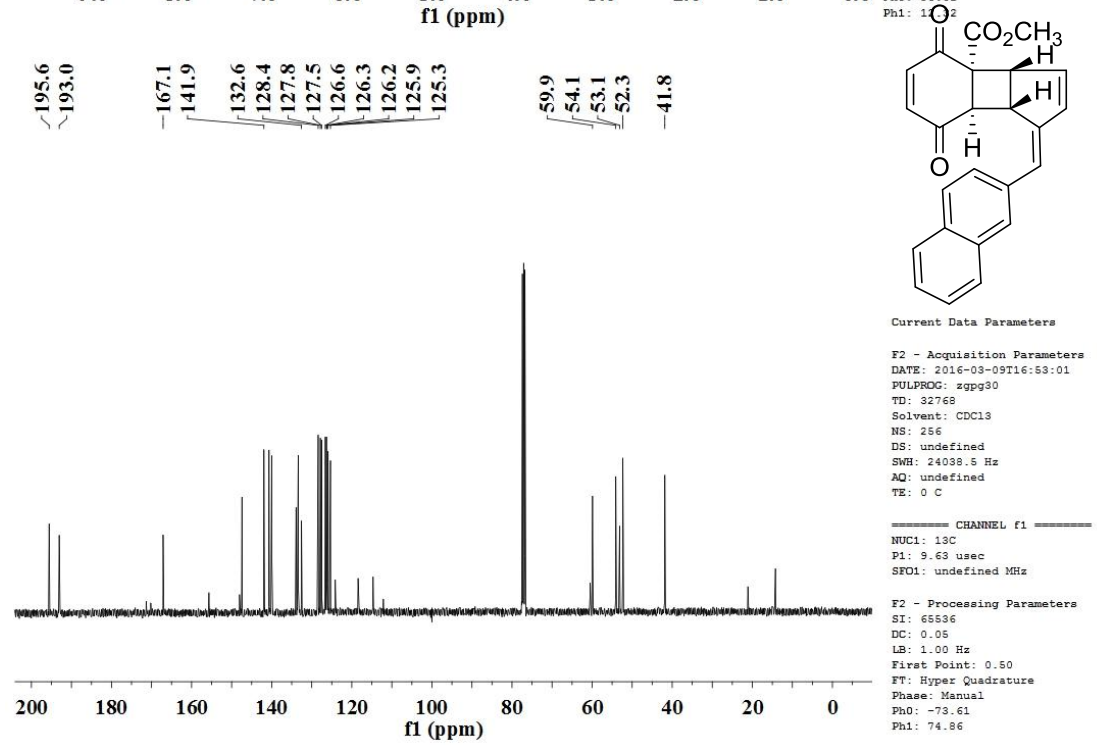
Current Data Parameters

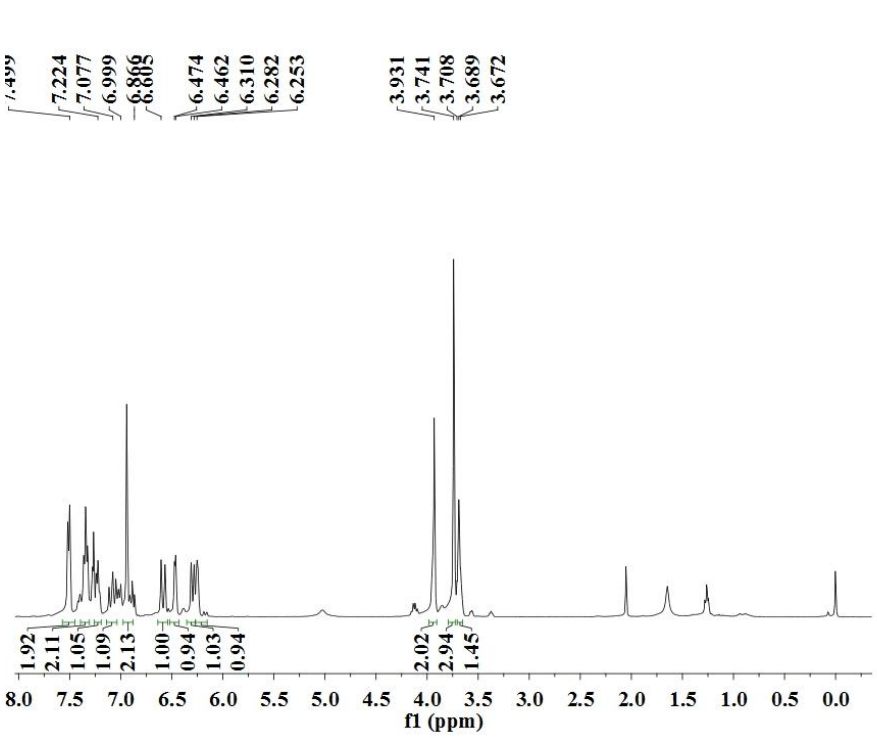
F2 - Acquisition Parameters
DATE: 2016-04-19T10:52:50
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 512
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C

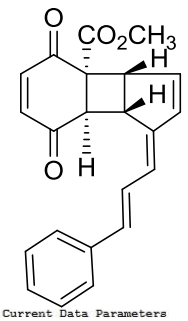
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -65.10
Ph1: 63.55


7.402
7.372
7.351
7.002
6.980
6.958
6.908
6.883
6.857
6.685
6.528
6.317
6.304
3.914
3.900
3.891
3.882
3.865
3.835
3.820
3.804
3.744
2.07
2.06
2.14
1.07
2.11
1.00
1.02
1.00
1.27
0.97
1.08
3.03
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)
O
CO2CH3
H
H
H
O
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2016-03-09T16:29:17
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 95.02


195.6
193.0
167.1
141.9
132.6
128.4
127.8
127.5
126.6
126.3
126.2
125.9
125.3
59.9
54.1
53.1
52.3
41.8
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)
O
CO2CH3
H
H
H
O
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2016-03-09T16:53:01
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -73.61
Ph1: 74.86



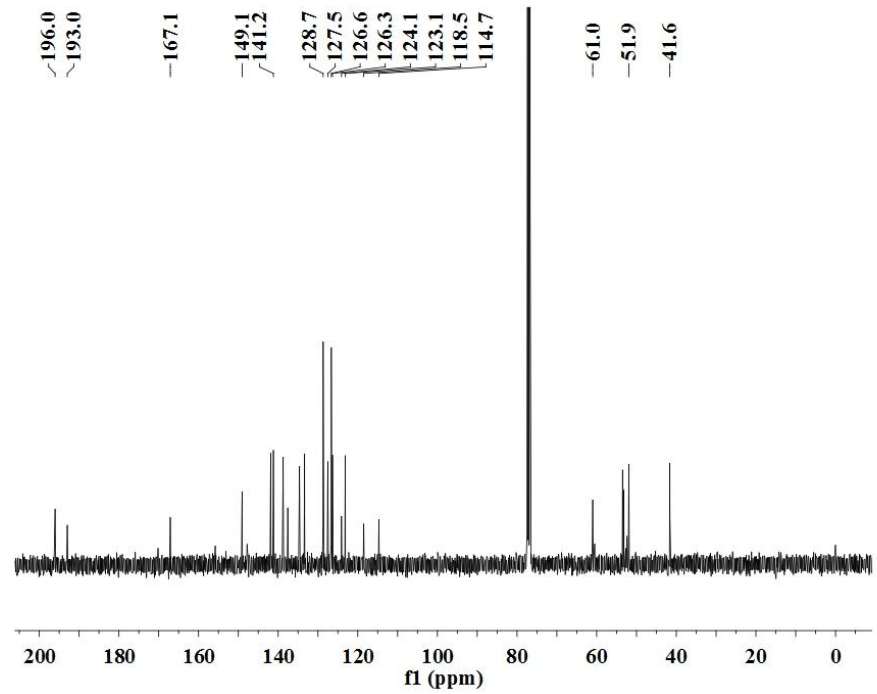


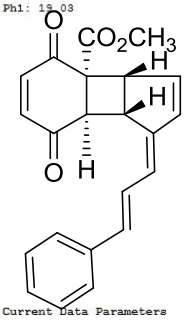
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-03-15T11:48:29
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 91.24
Ph1: 18.03



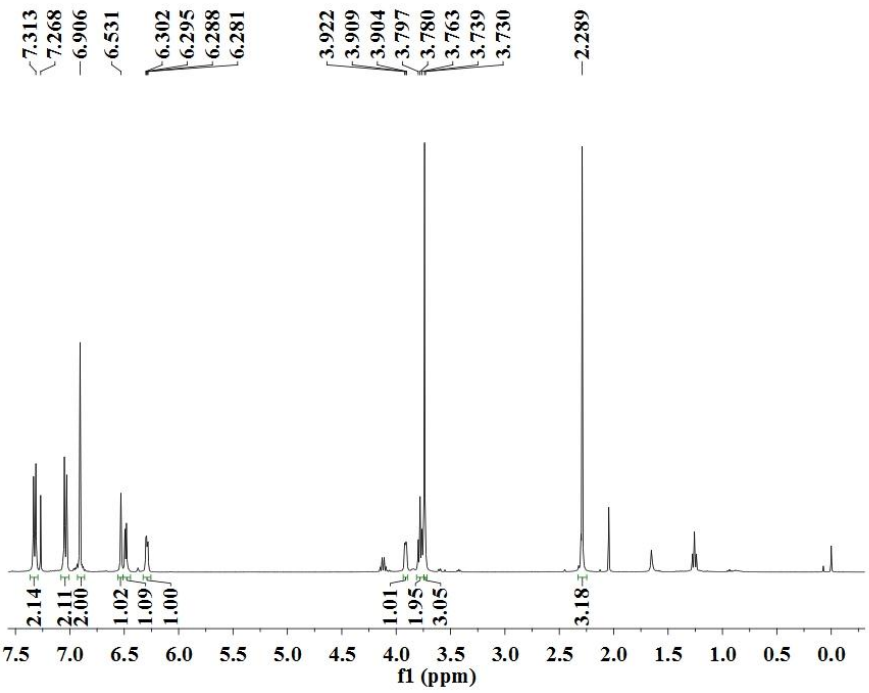


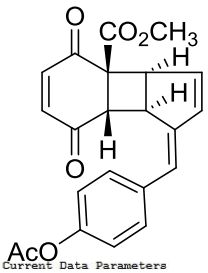
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-03-15T12:04:05
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -56.35
Ph1: 45.32



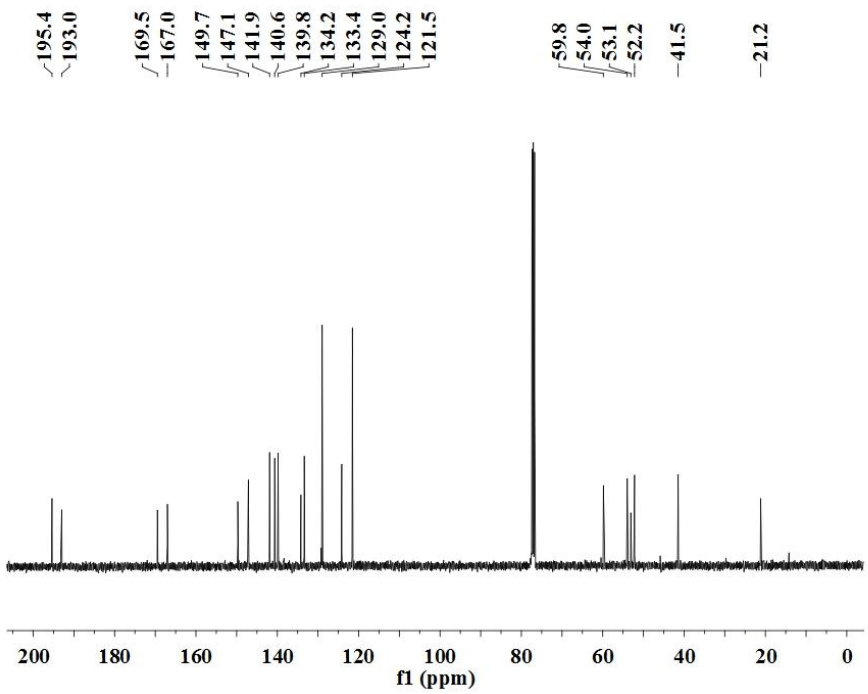


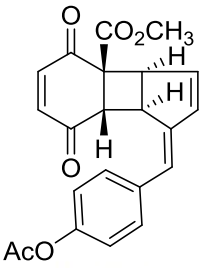
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-04-18T17:50:29
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 91.96
Ph1: 15.93



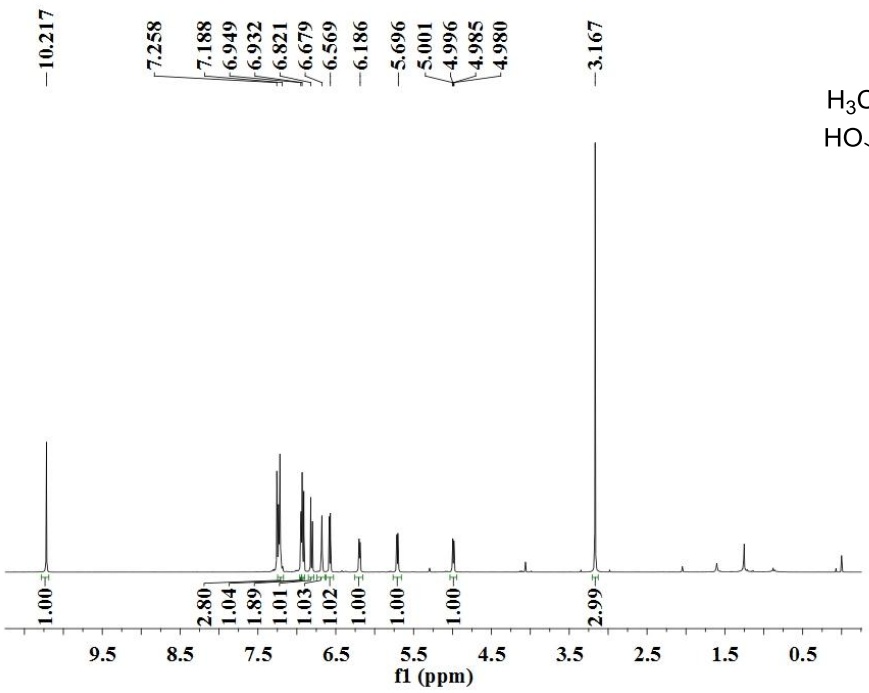


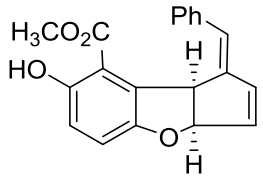
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-04-18T18:06:04
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -56.54
Ph1: 56.03



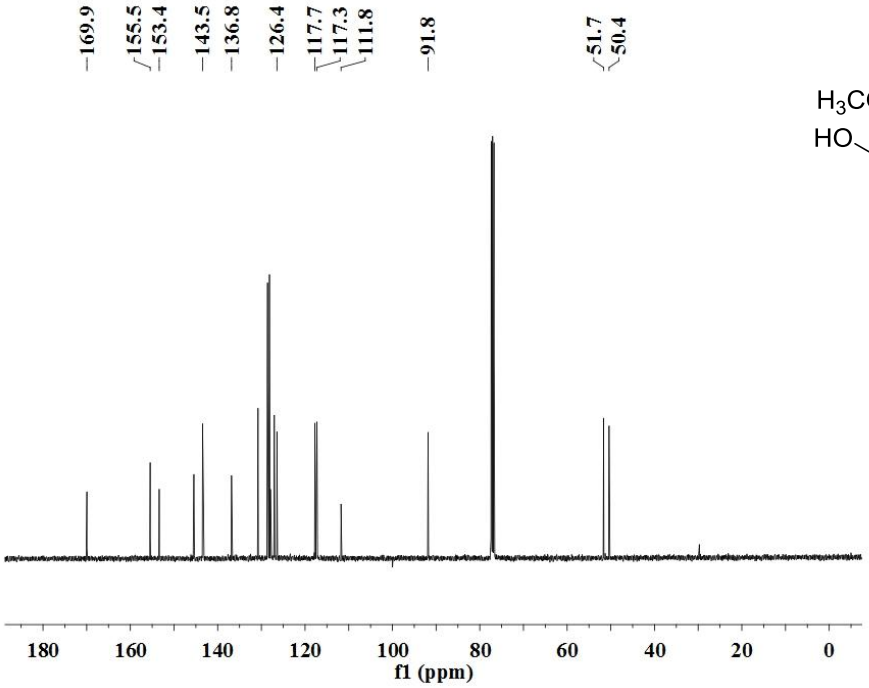


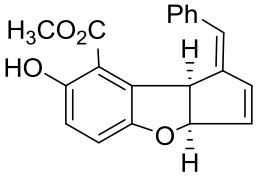
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-01-13T10:29:54
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -257.57
Ph1: 16.68



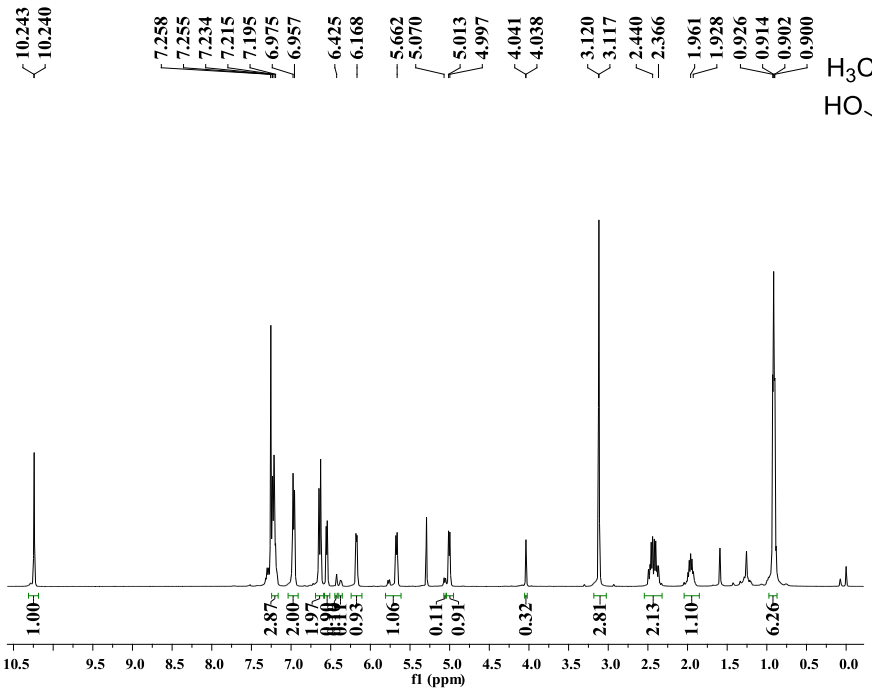


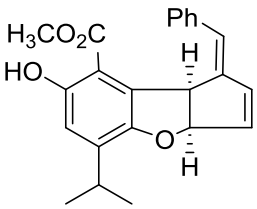
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-01-13T11:00:08
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 512
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -66.76
Ph1: 65.82



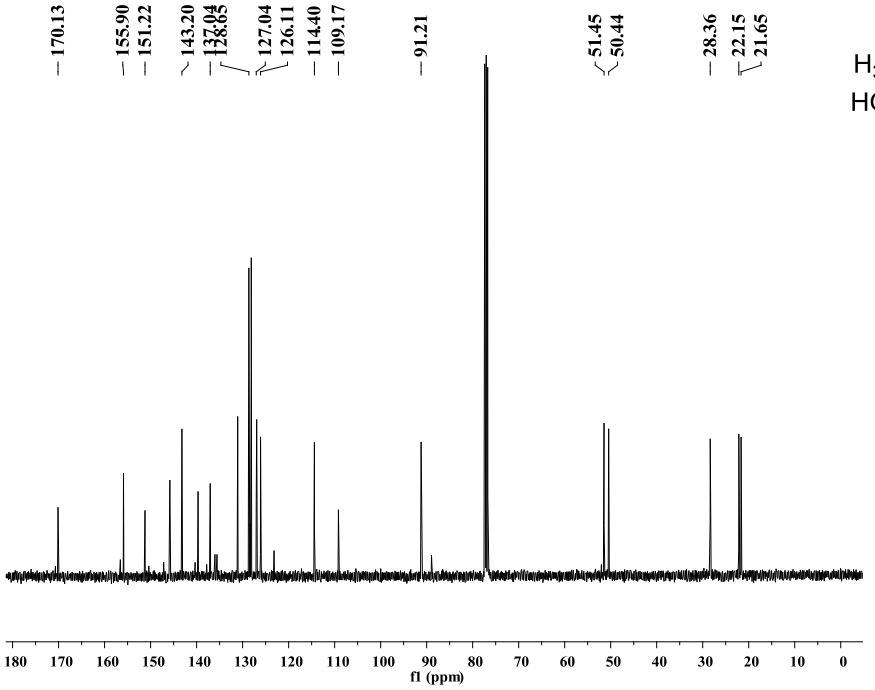


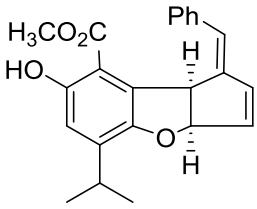
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2017-01-01T21:21:44
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.2 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -263.46
Ph1: 17.85



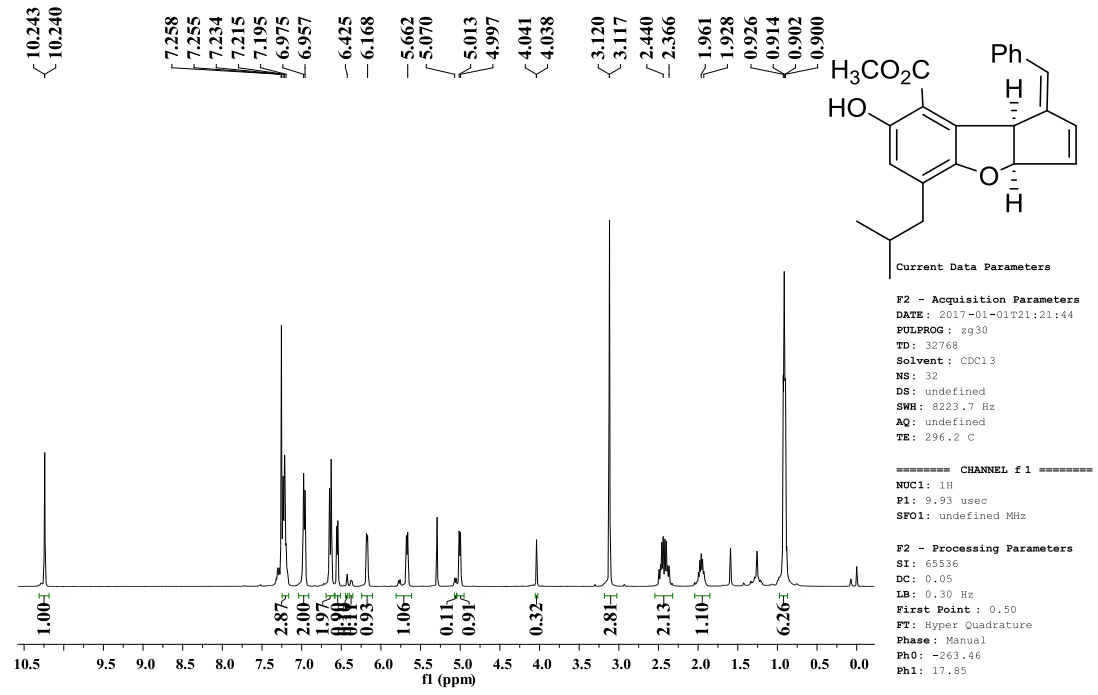


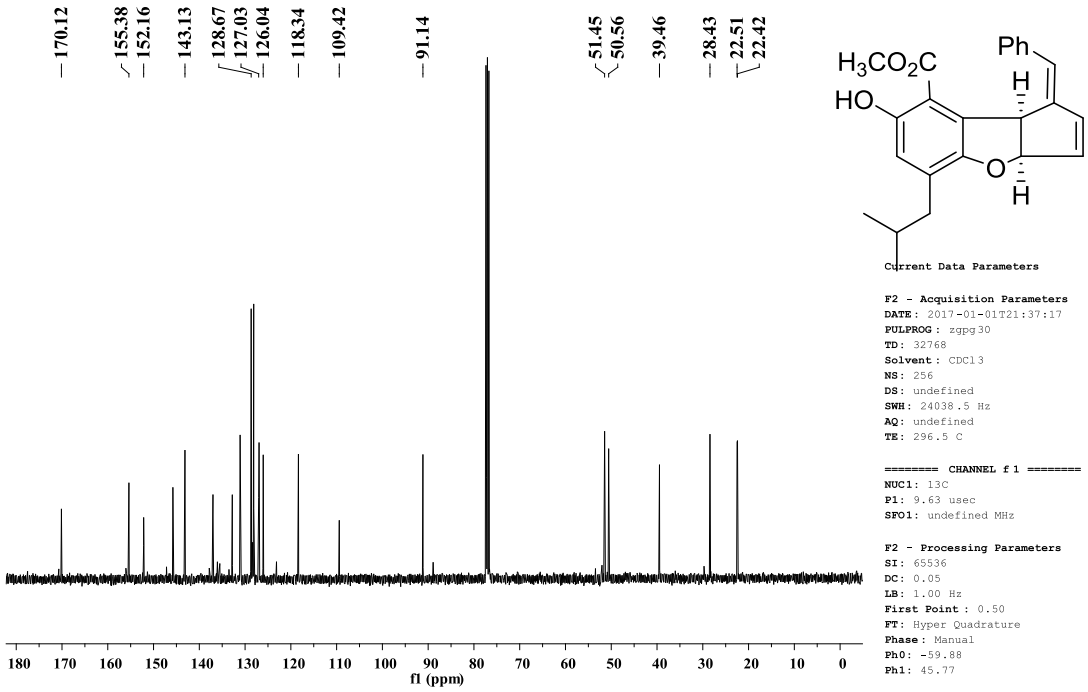
Current Data Parameters

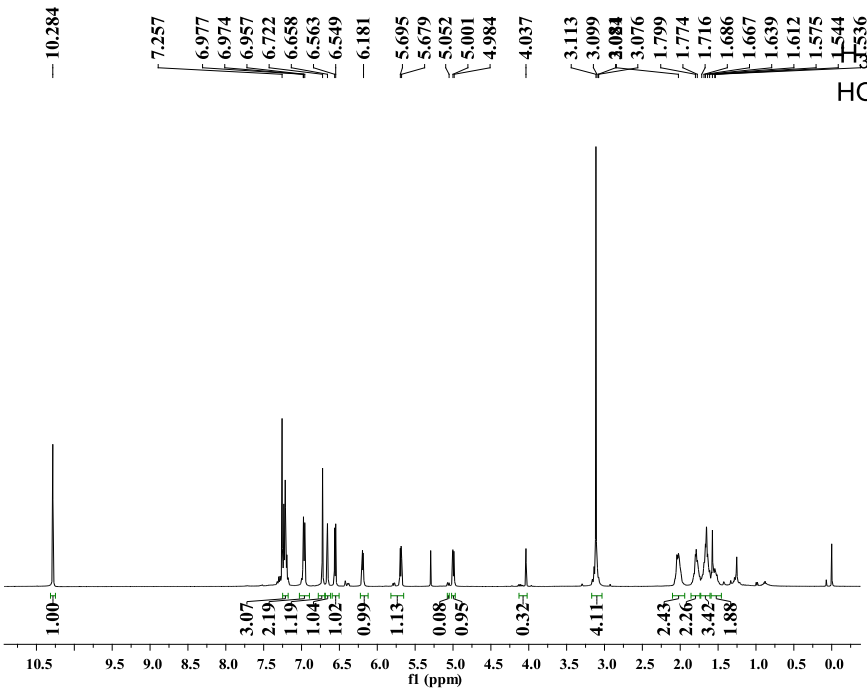
F2 - Acquisition Parameters
DATE: 2017-01-01T21:15:48
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.4 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -63.97
Ph1: 60.76


10.243
10.240
7.258
7.255
7.234
7.215
7.195
6.975
6.957
6.425
6.168
5.662
5.070
5.013
4.997
4.041
4.038
3.120
3.117
2.440
2.366
1.961
1.928
0.926
0.914
0.902
0.900
1.00
2.87
2.00
1.97
1.10
0.93
1.06
0.11
0.91
0.32
2.81
2.13
1.10
6.26
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
H3CO2C
HO
Ph
H
O
H
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2017-01-01T21:21:44
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.2 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -263.46
Ph1: 17.85


170.12
155.38
152.16
143.13
128.67
127.03
126.04
118.34
109.42
91.14
51.45
50.56
39.46
28.43
22.51
22.42
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
H3CO2C
HO
Ph
H
O
H
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2017-01-01T21:37:17
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.5 C
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -59.88
Ph1: 45.77

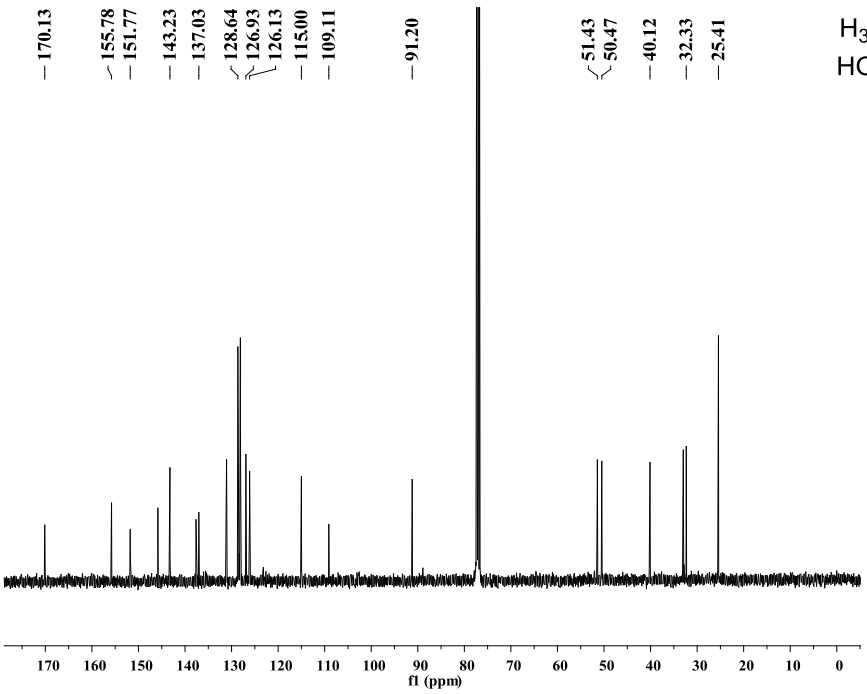


Current Data Parameters

F2 - Acquisition Parameters
DATE: 2017-01-01T21:42:56
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.1 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 98.97
Ph1: 8.14

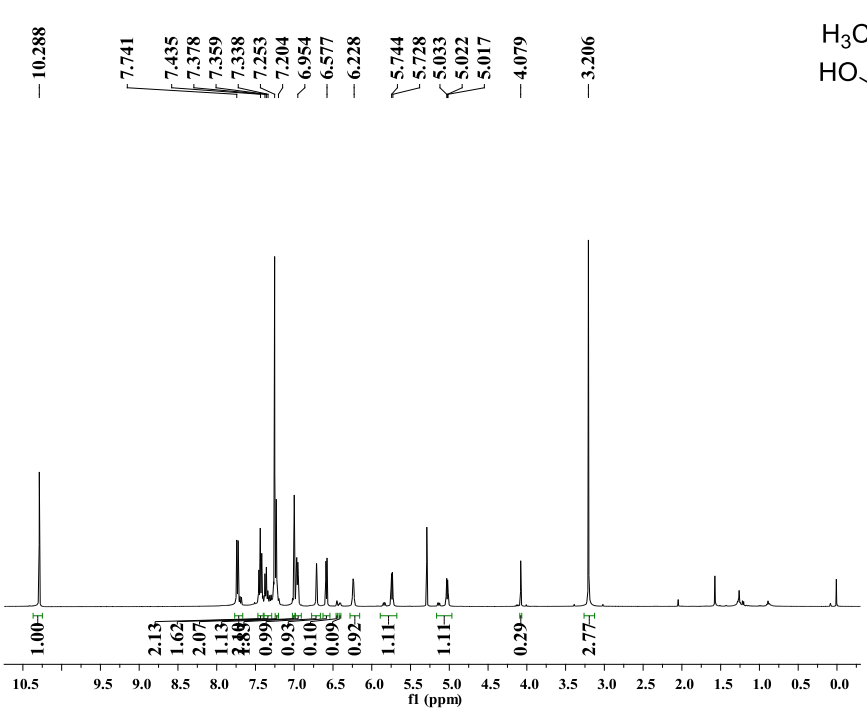


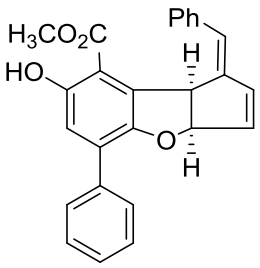
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2017-01-01T21:58:29
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.5 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -84.57
Ph1: 88.12



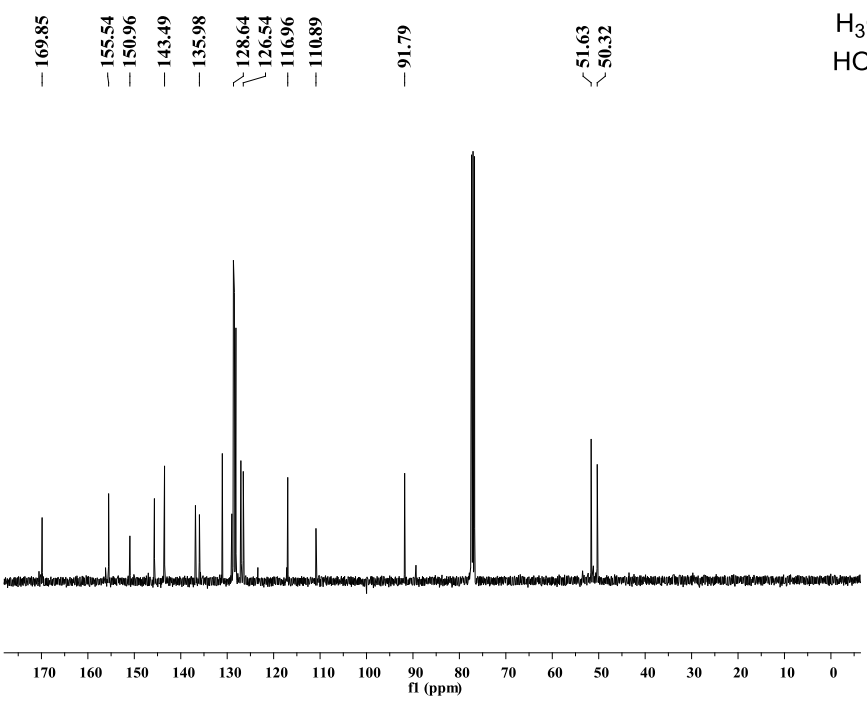


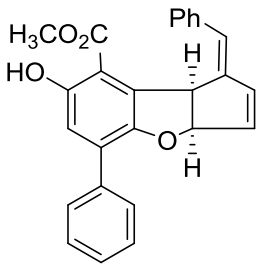
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2017-01-01T22:25:09
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296.2 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 101.30
Ph1: 1.07



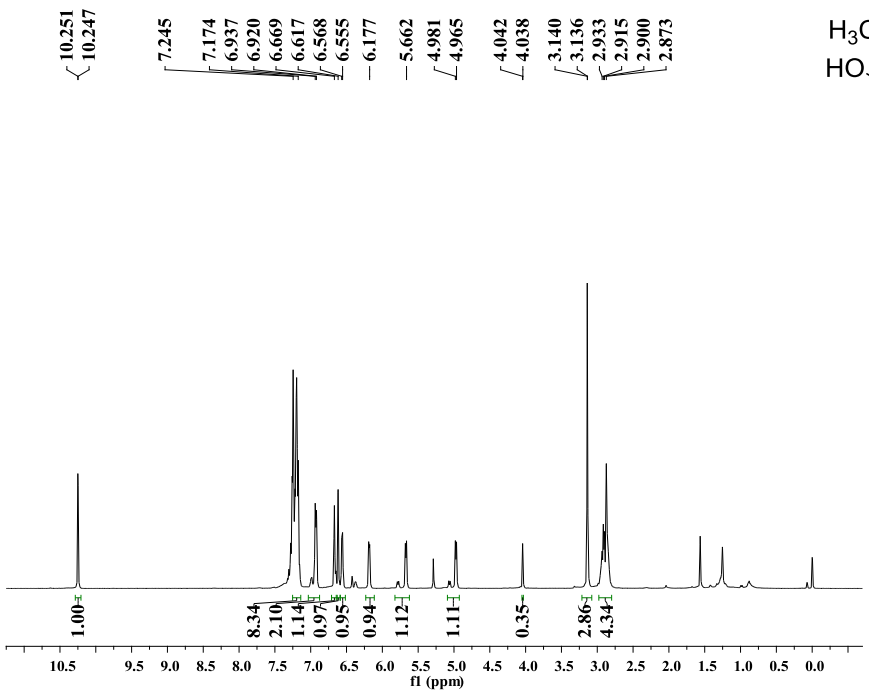


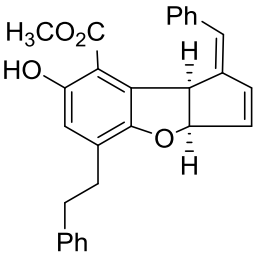
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2017-01-01T22:40:42
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.3 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -68.12
Ph1: 75.88



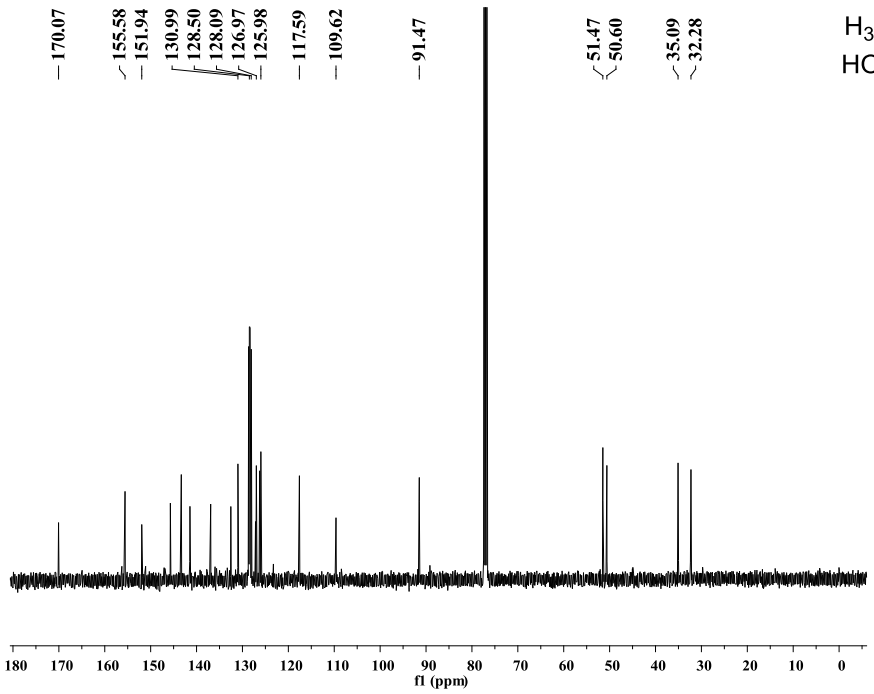


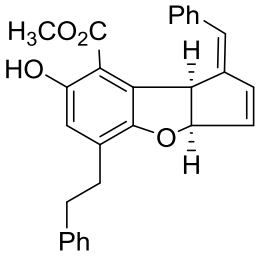
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2017-01-01T22:04:27
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 296 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 92.32
Ph1: 19.36



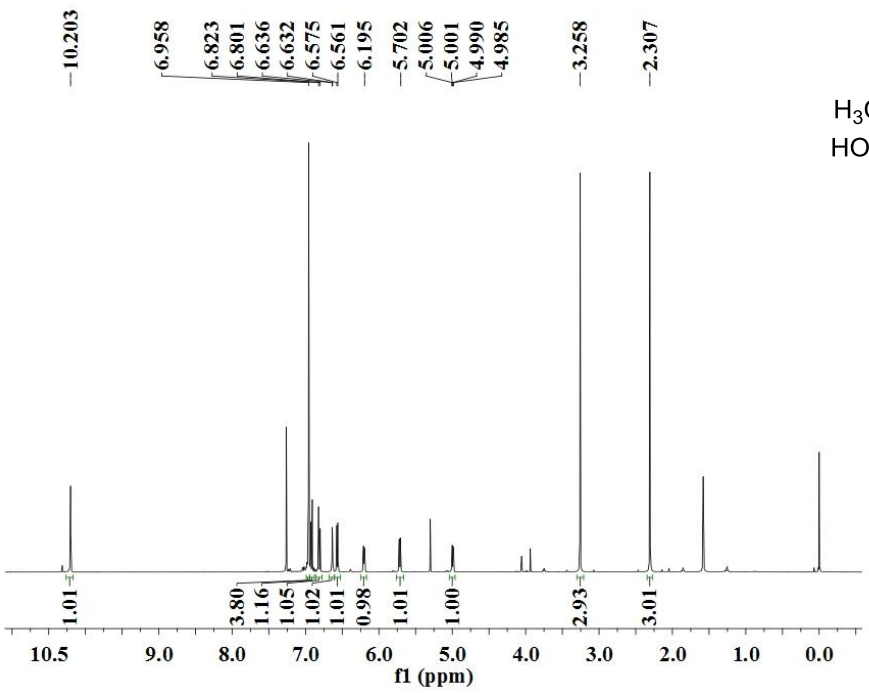


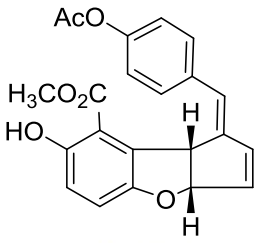
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2017-01-01T22:20:00
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 296.4 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -62.78
Ph1: 44.52



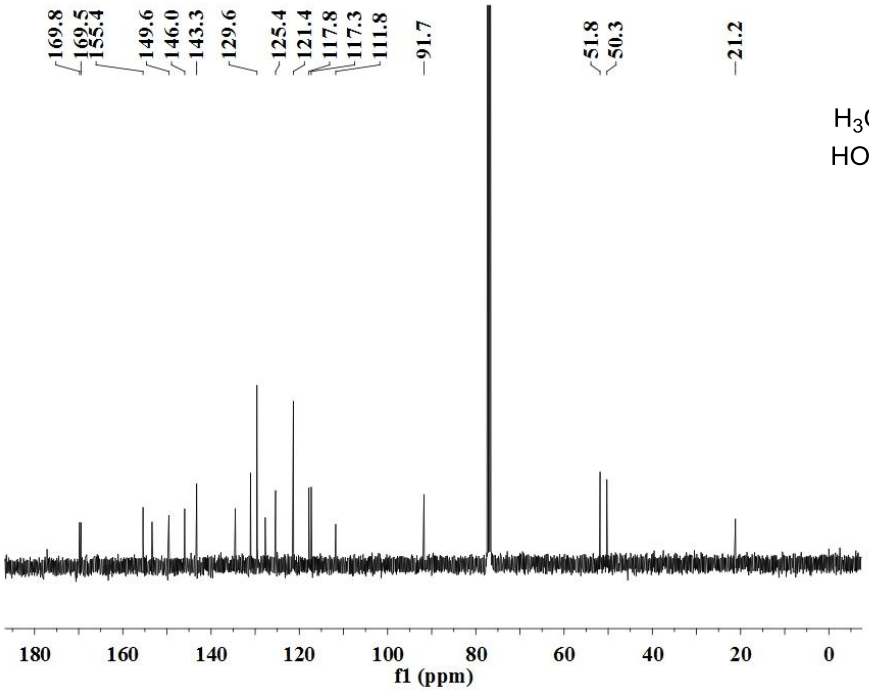


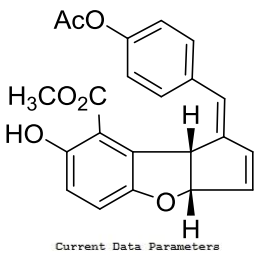
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-03-09T13:28:21
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 95.48
Ph1: 11.81



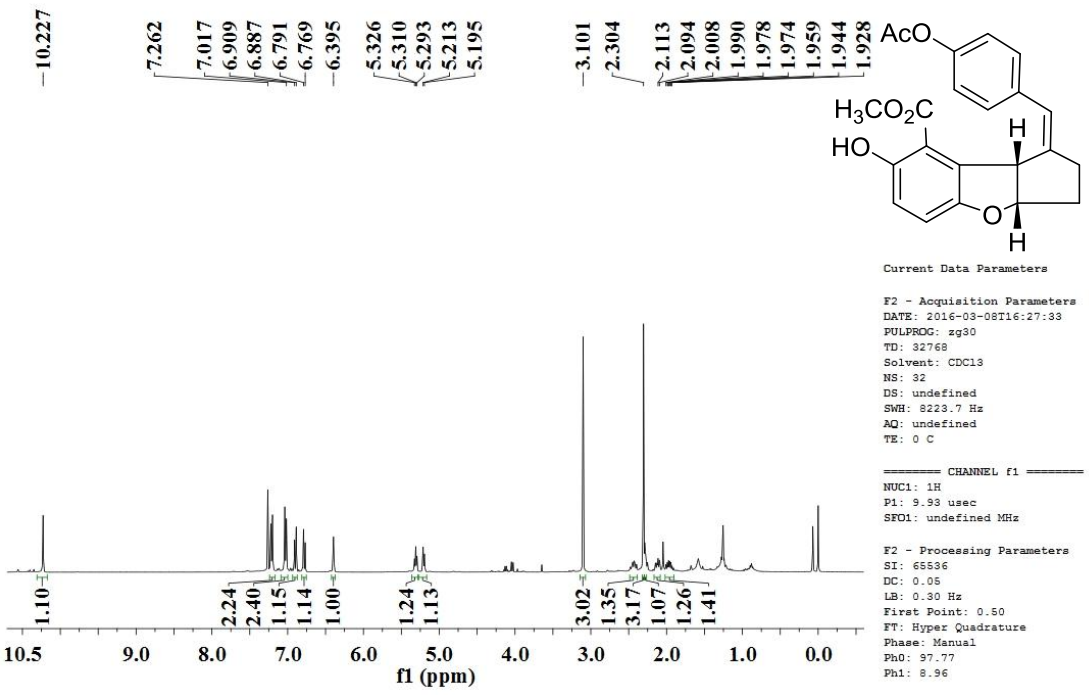


Current Data Parameters

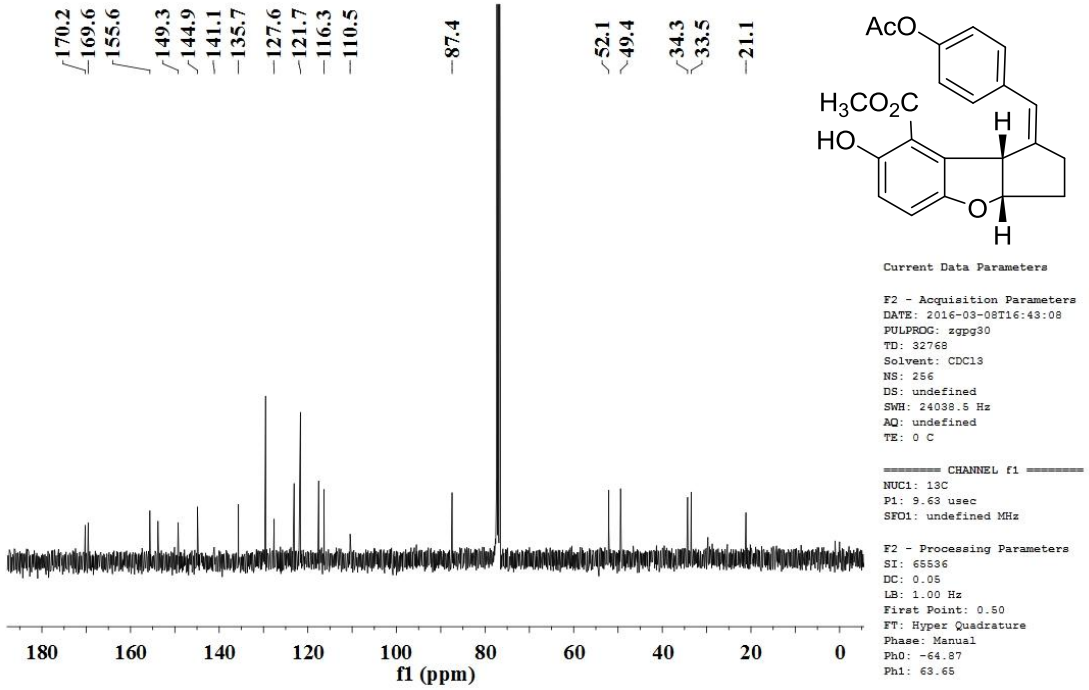
F2 - Acquisition Parameters
DATE: 2016-03-09T13:43:56
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C

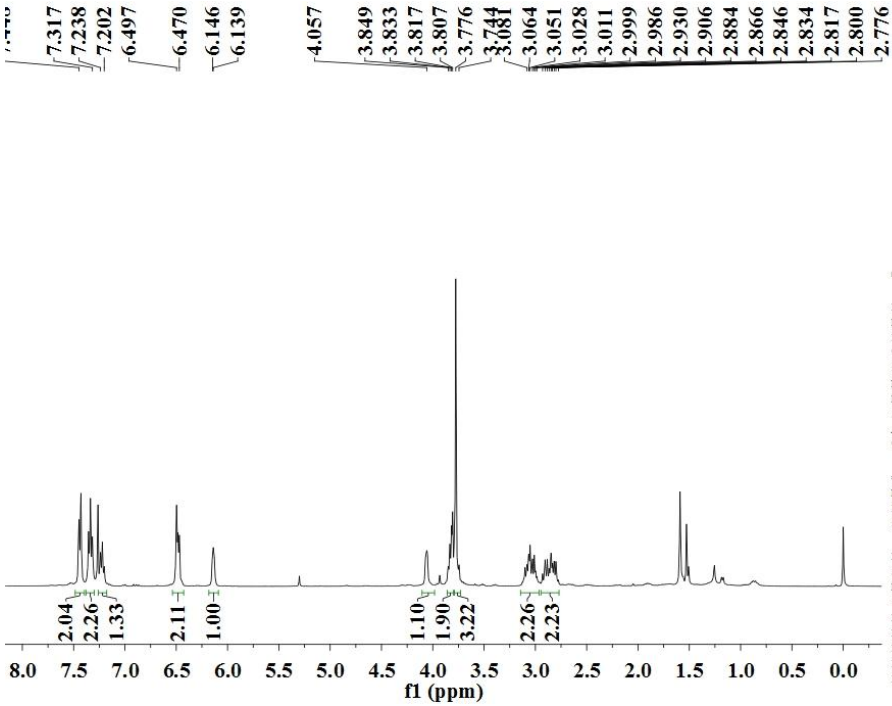
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -68.98
Ph1: 67.39


10.227
7.262
7.017
6.909
6.887
6.791
6.769
6.395
5.326
5.310
5.293
5.213
5.195
3.101
2.304
2.113
2.094
2.008
1.990
1.978
1.974
1.959
1.944
1.928
AcO
H3CO2C
HO
H
O
H
1.10
2.24
2.40
1.15
1.14
1.00
1.24
1.13
3.02
1.35
3.17
1.07
1.26
1.41
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2016-03-08T16:27:33
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: 97.77
Ph1: 8.96

.


170.2
169.6
155.6
149.3
144.9
141.1
135.7
127.6
121.7
116.3
110.5
87.4
52.1
49.4
34.3
33.5
21.1
AcO
H3CO2C
HO
H
O
H
180
160
140
120
100
80
60
40
20
0
f1 (ppm)
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2016-03-08T16:43:08
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -64.87
Ph1: 63.65

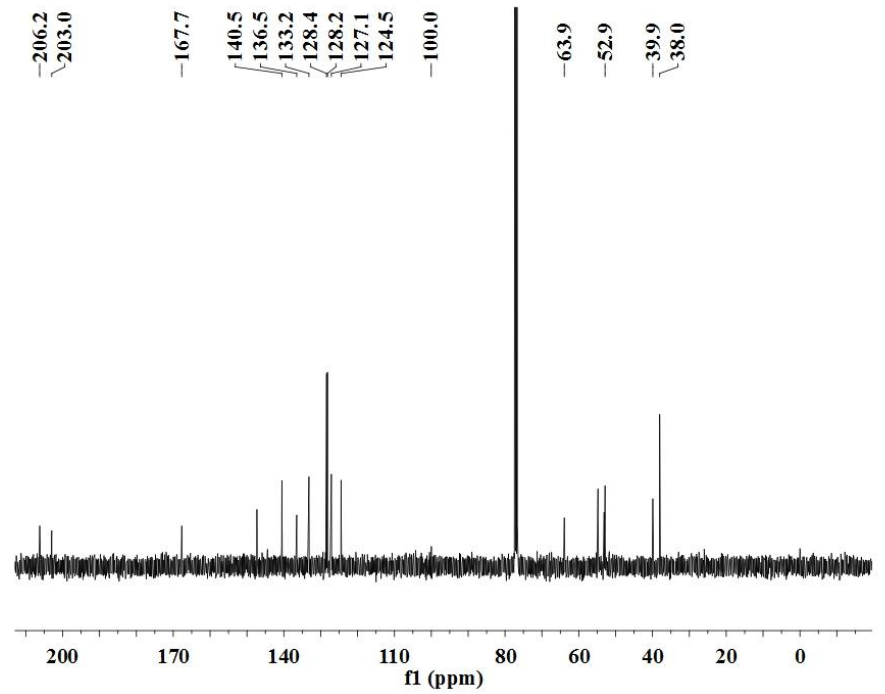


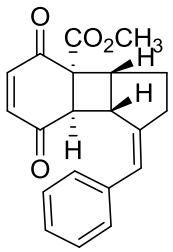
Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-03-23T10:08:59
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8223.7 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 1H
P1: 9.93 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -258.29
Ph1: 7.06





Current Data Parameters

F2 - Acquisition Parameters
DATE: 2016-03-23T10:24:35
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 0 C

======== CHANNEL f1 ========
NUC1: 13C
P1: 9.63 usec
SFO1: undefined MHz

F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -23.39
Ph1: -0.82